ANNUAL REPORT OF RESEARCH AND TECHNOLOGIC WORK ON COAL, FISCAL YEAR 1951

BY R. L. BROWN AND E. P. CARMAN

* * * * * * * * * * **Information Circular 7647**

UNITED STATES DEPARTMENT OF THE INTERIOR
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BUREAU OF MINES
J. J. Forbes, Director

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by

R. L. Brown[1/] and E. P. Carman[2/]

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INTRODUCTION AND SUMMARY

This report, summarizing the Bureau of Mines research and technologic work on coal and coal products for the period July 1, 1950, to July 1, 1951, is based largely on publications issued during the year. The sixteenth in an annual series, it gives footnote references to publications giving further details of the research and technologic work described, except in a few cases where research data presented herein has not been published previously.

During the year, the national fuels situation was generally one of plentiful supply.3/ Mineral-fuel resources appeared adequate for several hundred years at reasonably increasing rates of supply. Coal-production capacity was in excess of demand; petroleum supplies were adequate; and natural gas use was limited only by pipeline capacity. However, it was pointed out that a world war would drastically change this situation of relative plenty, particularly with regard to supplies of petroleum and its products. Conversion to the use of coal on a large scale would be required, and improved methods of mining and of processing and using coal in solid or fluidized form or converted to liquid fuels could materially contribute to our national safety.

Turning from the future, a historical review, prepared in connection with the 75th anniversary of the founding of the American Chemical Society, gave a graphic picture of the development of gas and fuel technology in this country from 1876 to 1951.4/ This development by industry, as well as by the Bureau of Mines and other research organizations, is the background for present Bureau of Mines research programs on coal that are directed toward providing the technical data necessary for the most-effective use of this abundant natural resource.

With the onset of the Korean situation and the stimulus of the national defense program, transportation difficulties and shortages developed, particularly of special coals and chemicals needed in the defense program.5/ These emphasized the need for continuing research and technologic development to provide the basic technical data needed for the most efficient development and proper conservation-in-use of our mineral-fuel resources. The Bureau of Mines research on the mining, preparation, and utilization of coal has been directed toward developing more complete information on the extent and quality of coal reserves, encouraging safer and more efficient mining practices, and preparing and utilizing coals to the best advantage, now and in the future.

3/ Fieldner, Arno C., The National Fuels Situation: Eng. Exp. Sta. News, Ohio State University, vol. XXIII, No. 1, February 1951, pp. 12-17, 38-42.

4/ Fieldner, A. C., Gauger, A. W., and Yohe, G. R., History of Gas and Fuel Chemistry in the U. S.: Ind. Eng. Chem., vol. 43, No. 5, May 1951, pp. 1039-1048.

5/ Boyd, James, Synthetics in Defense: Proc. Am. Petrol. Inst., vol. 30, sec. III, 1950, pp. 6-19.

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In December 1950 additional research facilities became available with the completion and occupancy of the new laboratory at Grand Forks, N. Dak., which was dedicated on September 29, 1951, as the Charles R. Robertson Lignite Research Laboratory. (See fig. 1.) Also, in December 1950, construction was started on a wing to the Anthracite Research Laboratory at Schuylkill Haven, Pa., to provide in this laboratory the space and facilities originally planned. (See fig. 2.) A garage-warehouse is also under construction.

To insure that coal purchased by the Government met guarantees, the Bureau continued its analytical services to Federal agencies in their purchases of coal by sampling and analyzing coal purchased on a guaranteed analysis basis. Consulting services were given Federal agencies on their fuel purchases. For this purpose, proximate, sulfur, heating value, ultimate, and other analyses were made on 10,850 samples of coal and coke from Government purchases and from tipple and breaker inspections. Analysis of 5,401 samples of coal-mine dusts enabled mine inspectors to determine the adequacy of rock dusting. Following its traditional pioneering assistance in the establishment of fuel standards, the Bureau cooperated with an Economic Commission for Europe committee working to establish an international standard coal-classification system. The Bureau's description of methods for analyzing coal and coke has been revised to incorporate recent improvements.

As part of the Bureau's cooperation under the Point-Four Program, technical assistance on coal mining and preparation was extended to South American countries.

In this country the investigation of known recoverable reserves of coking coal was continued in central and southern Pennsylvania, southern West Virginia, and eastern Kentucky to provide data on potential sources of metallurgical coke needed to maintain high rates of steel production. Recoverable reserves of 3,662 million tons of coking coal in beds 28-inches or more thick were shown for two counties in central Pennsylvania and one in eastern Kentucky. Studies of the preparation and carbonizing properties of coking coals in the eastern states were continued simultaneously with investigations of reserves of coking coals as the second and third phases of the coking-coal investigations. In the preparation phase the studies indicated which of the coals studied were suitable for metallurgical use or could be made so by conventional washing methods or special treatments. In the third phase the carbonizing properties and the yield and quality of coke and chemical products were determined for selected coals.

To provide data for development of safer and more efficient mining methods and equipment in the anthracite region of Pennsylvania, a German lightweight universal shearing machine was found adaptable to steep-pitch mining, and problems relating to roof control and to mine flood-water dangers were studied.

Commercial development of the first Diesel mine locomotive to be approved under Bureau of Mines Schedule 22 was brought much closer by tests on three units and the working out of necessary modifications with the manufacturers.

Great improvement in coal-mine safety in recent years was shown in studies of coal-mine explosions, fires, and fatalities, and the major causes and means of avoiding these accidents were pointed out. A schedule was developed for approval of respiratory protective devices, and arrangements were made for testing this type of equipment for Bureau approval. About 14,000 samples of mine air were analyzed to determine adequacy of coal-mine ventilation; over 1,600 dust-concentration and particle-size determinations were made; various types of electrical mine equipment were tested; and roof-bolting studies were made in the Bureau's continued efforts to increase mine safety and efficiency.

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Figure 1. - Charles R. Robertson Lignite Research Laboratory of the Bureau of Mines, Grand Forks, N. Dak.

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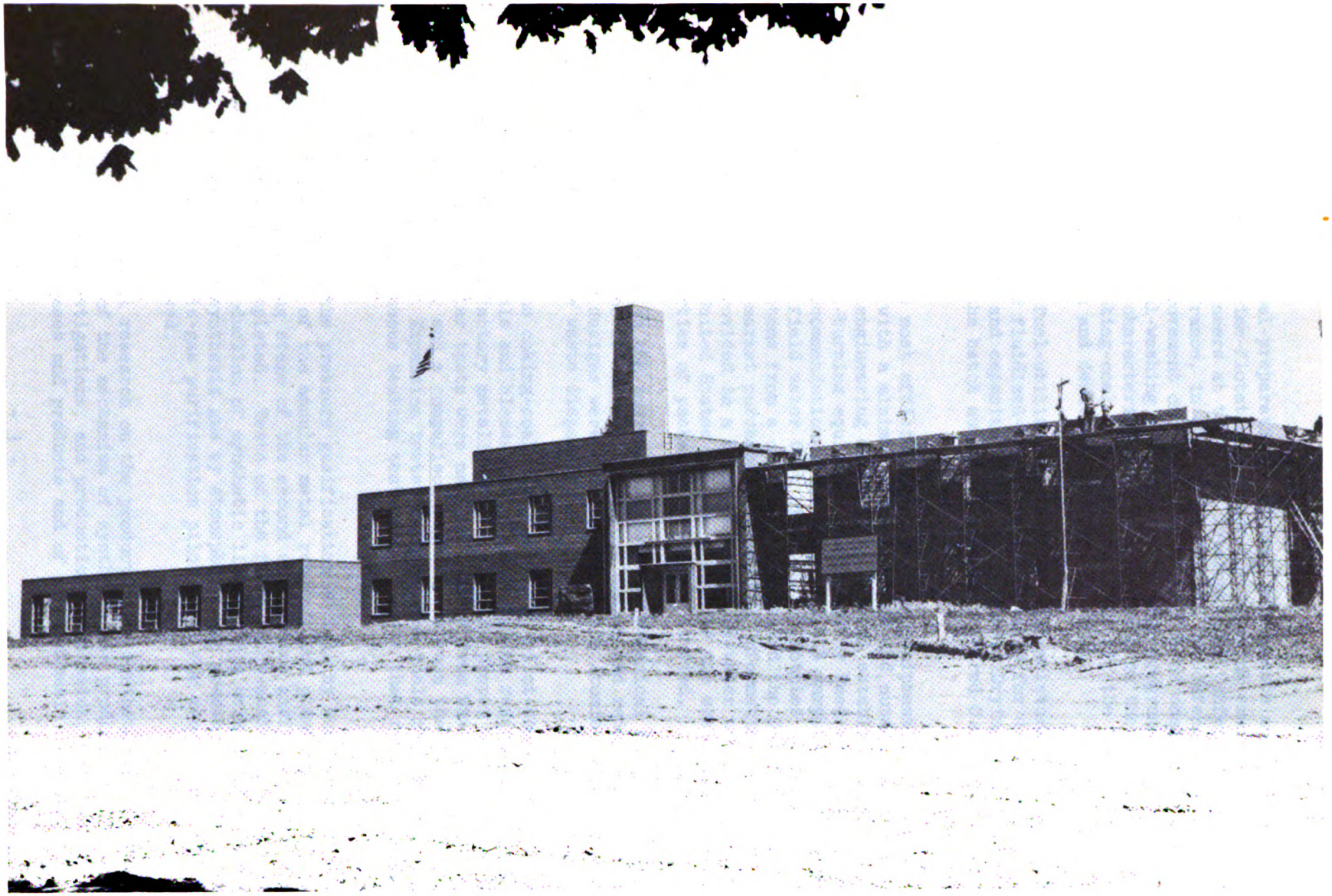

Figure 2. - Bureau of Mines Anthracite Research Laboratory, Schuylkill Haven, Pa.

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Methods of evaluating coal-preparation-equipment performance and a description of the Bureau-developed kerosine-flotation process for cleaning small sizes of coal were described by Bureau engineers at the First International Conference on Coal Preparation, held in Paris, France, in June 1950. To aid the defense program by providing information on improvement of coking coals and use of coal fines, major attention in the Bureau's coal-washing research and testing programs was focused on: (1) Study of the preparation characteristics of coking coals in the eastern States as the second phase of the coking-coal investigations, and (2) the testing and improving of equipment to clean and dewater fine sizes of coal.

Research to improve the fuel-utilization value of high-moisture western coals indicated that the continuous fluidized-bed dryer is suitable for drying any noncoking fuel, and equipment size and capacity can be calculated. Drying lump lignite with high-pressure steam in the batch autoclave process required 0.8-pound steam per pound of water removed.

To aid in purchasing the most efficient fuel-burning equipment and in obtaining maximum operating efficiency, with a minimum of maintenance and outage, the Bureau continued to give consulting engineering services to Federal establishments in selecting, testing, and use of fuel-burning equipment and in boiler feed-water conditioning and testing. Analyses and recommendations were made on 9,714 samples of boiler water. Distribution of 1,148 Bureau field water test kits, 12,764 bottles of reagents, and 14,781 test-kit-replacement items from a central source saved the Government about $20,000 over individual open-market purchases. Essential information needed by boiler-plant operators was provided in a handbook on boiler-water treatment. Air-pollution prevention in the United States and the growth of the western coal industry were reviewed, and the properties of packaged fuels were studied.

The Bureau cooperated with the American Society of Mechanical Engineers in heat-absorption studies in large power boilers to improve design and reduce fuel consumption; studies of incinerator designs were made to assist the Atomic Energy Commission in the problem of radioactive waste disposal.

As the third phase of the coking-coal investigations, pilot-scale coal-carbonization tests were made on coals and blends from 15 Appalachian region coal beds. Most of the coals made satisfactory metallurgical coke with proper blending and carbonizing temperatures. Similar tests were made on 2 coals from Washington and 14 from Columbia, South America, and a comparison was made between the BM-AGA and the experimental slot-type oven. Expansion tests indicated the coals that might injure commercial ovens, and spontaneous heating tests showed coals that require special attention in storing.

Results of research on the pressure gasification of coal by the Lurgi process were reported, and operation of the annular metal retort pilot plant for gasifying lignite continued. The firing stage of the second underground gasification experiment at Gorgas, Ala., was completed. Tests on the first pilot-scale gasifier to produce synthesis gas for production of synthetic liquid fuels were completed, and new pilot plants to produce synthesis gas by atmospheric and pressure gasification were constructed. A synthesis-gas purification pilot plant for removal of both H_2S and CO_2 was tested and improved.

The Bureau's fundamental research on the production of synthetic liquid fuels was continued, with studies of the mechanism of synthesis-gas production, hydrogenation of coal, catalyst investigations, and production, separation, and identification of coal-hydrogenation gases and products and of gas-synthesis products.

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The Coal-Hydrogenation Demonstration Plant at Louisiana, Mo., completed several successful liquid- and vapor-phase tests, and much of the gasoline produced has been used by the Army in transport vehicles. The Gas-Synthesis Demonstration Plant and a new vertical coal-dust gasifier were completed, and all units of the system were individually tested. Detailed cost estimates were prepared for planning commercial synthetic fuel plants to suit specific fuel and other conditions as interest mounted in the economic aspects of the production of synthetic liquid fuels and coal chemicals from coal by hydrogenation and by gas-synthesis processes.

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Old Ben Coal Corp.
Philadelphia & Reading Coal
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Texas Power & Light Co.
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ORIGIN, COMPOSITION, AND PROPERTIES OF COAL

Inspection, Sampling, and Analysis

Production of coal during the fiscal year 1951 was equal to or in excess of demand, and Federal agencies had no difficulty in obtaining bids covering their requirements. All agencies were able to make their purchases on a guaranteed analysis basis, including penalties for delivery of substandard coal. The Navy Department purchased coal for the Army, Air Force, and Navy and is the largest Federal purchasing agency of coal. In general, other departments purchase their own coal supply, except that the Federal Supply Service purchases the supply for some agencies whose requirements are small. Contracting for coal for the fiscal year 1952 started in March and continued through the balance of the fiscal year 1951.

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Coal requirements of Federal agencies for the fiscal year were estimated at approximately 5,200,000 tons. Requests from Federal agencies for analyses for use in evaluating bids continued at about the same rate as the previous year, which was the largest in history. This was caused by strict adherence to specification requirements that eliminated mines on which there are no Bureau of Mines analysis records.

Urgent requests from Federal agencies, mine operators, and coal-sales agencies for service in obtaining mine tipple analyses continued throughout the year. A total of 1,521 tipple samples was collected at 549 mines in 17 States. At the beginning of the year, requests for sampling 350 mines were on file; although additional requests were received daily, the number of requests on file at the close of the year had been reduced to about 300. For carbonization, grindability, hydrogenation, electrode carbon, and other special tests, 602 samples were collected at 539 mines.

At the request of the Navy department, review and recommendations for improving sampling methods were made at one station, and recommendations for sampling equipment and establishing a sampling system were made for another.

Proximate, sulfur, heating value, ultimate, and other analyses were made on 10,850 samples of coal and coke from Government purchases and from tipple and breaker inspections. About two-thirds of these samples came from branches of the Department of National Defense, the rest from civilian agencies of the Government. In addition to ash-fusion temperature determinations on 3,494 samples and Hardgrove grindability indexes on 443 samples, free-swelling indexes were reported on 772 samples at the request of the Army for information on the caking properties of coal, since the Army was considering incorporating this item in their future coal specifications.

To insure that adequate rock dust was being used in coal mines to prevent propagation of coal-dust explosions, the Bureau's coal-mine inspectors submitted 5,401 samples of road, roof, rib, and gob dusts, representing 1,235 mine inspections. As the analyses are used for making recommendations on safety, the samples were in most cases received, analyzed, and reported the same day.

A total of 20,658 samples of coal, coke, char, tar residues, coal-mine dusts and other carbonaceous materials was analyzed during the year, requiring 268,116 chemical and physical tests.

Analyses of Miscellaneous Materials

As a further aid in maintaining safe and healthful conditions in coal mining, laboratory examination of 20 samples of limestone and 1 of dolomite showed these materials were suitable for rock-dusting coal mines to prevent coal-dust explosions, but 3 other materials were found unsuitable. To assist in making recommendations for improving feed-water treatment at Government steam plants, 12 boiler-water scales or deposits and 7 boiler compounds were analyzed. For use in research problems in the Bureau relating to coal carbonization, gasification, and combustion and production of synthetic liquid fuels, chemical constituents were determined for 66 samples of coal ash and slag, fly ash, and related materials, including a material claimed to be a fuel saver and 2 soot removers.

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Constitution, Properties, and Analytical Methods

Coal Classification

Increased world trade in coal since World War II has shown the need for international methods of analyzing, testing, and classifying coals to provide a common language for evaluating coals of various countries. The Bureau of Mines is cooperating with the Classification Working Party of the Coal Committee, Economic Commission for Europe, in working toward an international standard classification system. A report was prepared for the committee discussing the relationship of the free-swelling index to volatile matter on the dry, ash-free basis for certain caking coals of the United States. It was shown that the free-swelling index alone is not a significant indicator of rank classification for American coals. (See fig. 3.)

Petrographic Studies of Coal

A complete petrographic study, including thin section and broken coal analysis, was made of a column sample representing approximately 8 feet of coal from the Illinois No. 6 bed, Darmstadt No. 1 mine, St. Clair County, Ill., collected for laboratory hydrogenation tests. The thin-section analysis reveals a bright-type coal, which, except for mineral matter, is rather uniform throughout the bed. The coal is composed predominantly of anthraxylon, as shown in table 1.

TABLE 1. - Petrographic composition of coals from No. 6 bed, Illinois, and Coalburg bed, West Virginia, as-received basis

	No. 6 bed, percent	Coalburg bed, percent
Anthraxylon	65.0	17.4
Translucent attritus	24.7	43.1
Opaque attritus	2.7	34.1
Fusain	2.9	1.9
Translucent mineral matter	2.8	3.5
Pyrite	1.9	-

A complete thin-section analysis was made of a column sample of the Coalburg bed from the Silush mine, located in Boone County, W. Va. The microscopic analysis revealed that this coal contained 68 percent splint and 32 percent semisplint. It had the highest percentage of splint coal of any sample yet examined in this laboratory. The high-percentage opaque matter and fusain in this coal, as shown in table 1, would probably exclude its use for coking or hydrogenation purposes.

A discussion of certain geological and petrographic aspects of American lignites, developed in an investigation of solvent extractable waxes from domestic lignites, was published.6/ In the Gulf Coastal Plain area of the States of Alabama, Mississippi, Tennessee, Arkansas, Louisiana, and Texas, numerous deposits of lignite, mostly local in extent, are found in the Wilcox formation of the Lower Eocene age. The Northern Great Plains States of South Dakota, North Dakota, Montana, and Wyoming are underlaid with rather extensive blanket-like beds of lignite occurring in the Fort Union formation, which is of the Paleocene age. Along the Pacific coast area of northern California, Oregon, and Washington, there are spotty occurrences of lignite, which are of the Eocene age.

6/ Parks, B. C., Petrography of American Lignites: Econ. Geol. vol. 46, No. 1, January-February 1951, pp. 23-50.

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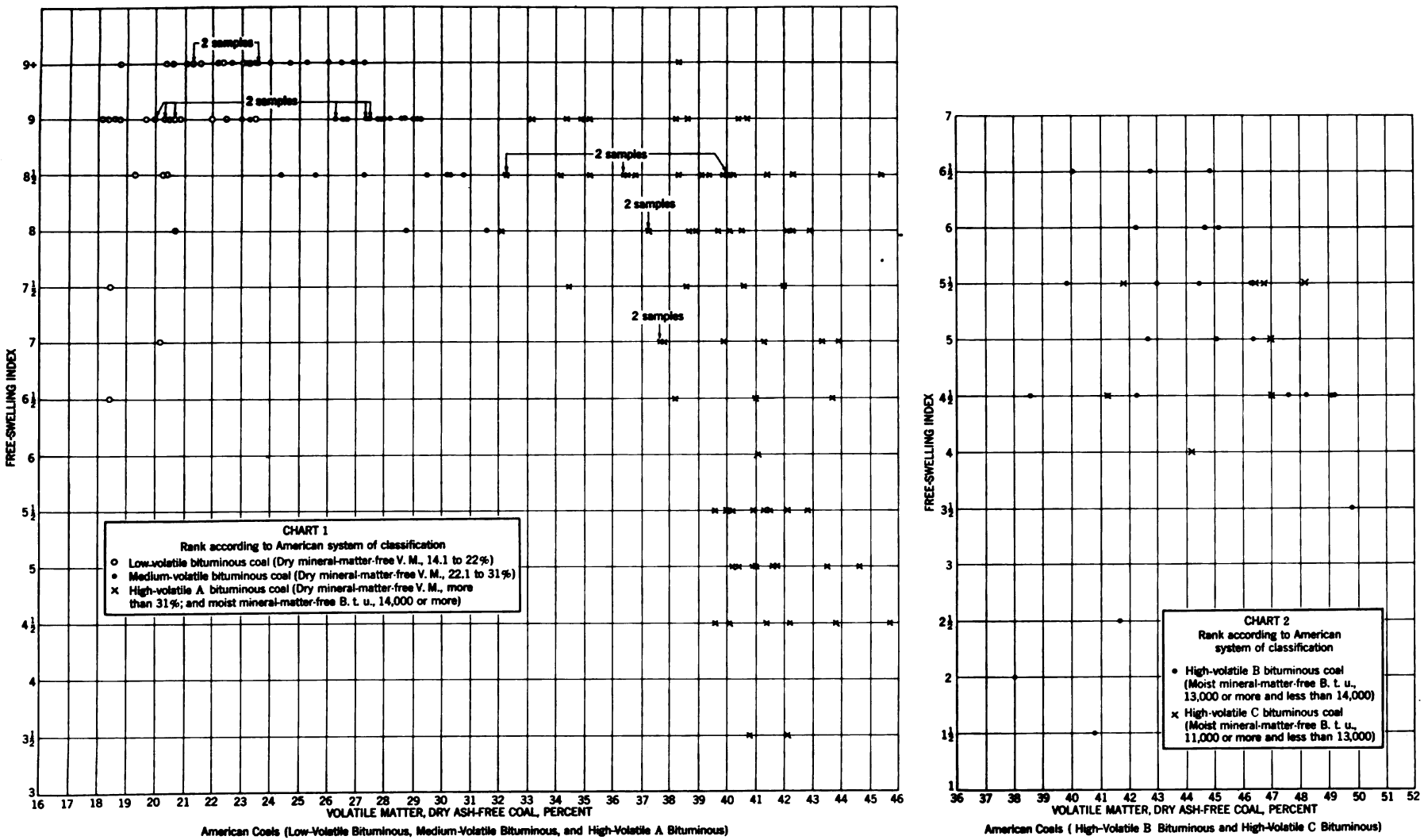
2 samples
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FREE-SWELLING INDEX
VOLATILE MATTER, DRY ASH-FREE COAL, PERCENT
CHART 1
Rank according to American system of classification
Low-volatile bituminous coal (Dry mineral-matter-free V. M., 14.1 to 22%)
Medium-volatile bituminous coal (Dry mineral-matter-free V. M., 22.1 to 31%)
High-volatile A bituminous coal (Dry mineral-matter-free V. M., more than 31%; and moist mineral-matter-free B. t. u., 14,000 or more)
American Coals (Low-Volatile Bituminous, Medium-Volatile Bituminous, and High-Volatile A Bituminous)
FREE-SWELLING INDEX
VOLATILE MATTER, DRY ASH-FREE COAL, PERCENT
CHART 2
Rank according to American system of classification
High-volatile B bituminous coal (Moist mineral-matter-free B. t. u., 13,000 or more and less than 14,000)
High-volatile C bituminous coal (Moist mineral-matter-free B. t. u., 11,000 or more and less than 13,000)
American Coals (High-Volatile B Bituminous and High-Volatile C Bituminous)

Figure 3. - Free-swelling index of American low- and medium-volatile and high-volatile A bituminous coals and American high-volatile B and C bituminous coals as a function of percent volatile matter.

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Petrographic variety	Color variation	Profile section (Megascopic)	Megascopic description (Surface examination)	Microscopic description (Thin section examination)	Typical quantitative determinations of petrographic composition. (Thin section analysis)	Typical photomicrographs	Magnification	Typical solvent extraction yields. Benzene	Typical solvent extraction yields. Benzene-alcohol 80-20%
Xyloid	Brown to dark brown		Relatively hard, firmly consolidated, banded coal. Made up of alternating layers and lenses of smooth, semi-bright, and of dull, grainy coal. Occasional thin layer and lens of black, soft fusain. Most layers and lenses of all components are less than 1/4 inch thick. Occasional thick "woody" layers easily recognizable with unaided eye to be of wood derivation. Brownish streak. Wet surface, black; dry, dark brown.	Orange translucent bands and lentils of anthraxylon, the predominant coal component present. Anthraxylon commonly shows distinct cellular structure of woody tissue in transverse or longitudinal cross sectional view. Dark red inclusions common in some anthraxylon bands. Attritus contains opaque matter and particles of fusinite, but consists mostly of brownish red humic degradation matter in very fine shreds and particles. Yellow translucent attritus of minor importance. Occasional thin layers and lenses of fusain showing woody structure and mineralization.	Anthraxylon 61% Translucent attritus 27 Opaque attritus 9 Fusain 3		65x 35x 65x 335x	N. Dak 1.2 Texas 2.0	2.8 4.2
Attrital	Brown to dark brown		Relatively hard, compact non-banded coal. Surface dull and grainy textured. Occasional thin layers and lenses of smooth textured semi-bright coal. Brownish streak. Wet surface, dark brown; dry, brown.	Consists mostly of brownish red humic degradation matter with thin layers and lenses of anthraxylon. Opaque attritus and fusinite common. Occasional layers and lentils of fusain. Yellow translucent attritus in subordinate amounts.	Anthraxylon 19.6% Translucent attritus 80.0 Opaque attritus .4 Fusain .0		50x	Ark. 7.6 Calif. 9.5	11.6 13.6
	Light brown		Tough, compact, non-banded coal. Surface dull and grainy. Occasional thin layer and lens of smooth textured coal. Occasionally particles of yellowish resin visible on surface. Slick "oily" trace on freshly cut surface. Light brown streak. Wet surface, brown; dry, light brown.	Brownish red humic matter and yellow translucent attritus with occasional thin layers and lentils of anthraxylon. Opaque matter of minor importance. Fusinite particles and fusain layers and lentils rare. Yellow translucent attritus very finely divided - particle size dominately less than 30 microns. Few large (30-80 microns) resinous bodies.	Anthraxylon 9.6% Translucent attritus 89.1 Opaque attritus .4 Fusain .0		50x	Ark. 8.9 Calif. 16.8	13.6 21.3
	Light brown		Tough, compact, non-banded coal. Surface dull and grainy. Occasional thin layer and lens of smooth textured coal. Grainy particles of resin common. Fragments of lump-type resin several millimeters in size occasionally found. Light brown streak. Wet surface, brown; dry, yellowish brown. Slick "oily" trace on freshly cut surface.	Brownish red humic matter and yellow translucent matter with occasional anthraxylon layer and lentils. Opaque matter and fusinite rare. Abundance of coarse yellow translucent attritus. Round resinous bodies and cuticular material prominent, and relatively of large size.	Anthraxylon 8.1% Translucent attritus 91.6 Opaque attritus .3 Fusain .0		35x	Ark. 10.2	15.7

Figure 4. - Petrographic descriptions and solvent extraction yields of different varieties of American lignite.

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The important deposits of lignitic coals in the United States are classed as lignite according to the American Society for Testing Materials system of classification by rank, but there are a few scattered, small deposits that would be classified as brown coal. These brown coals are of much later geological age, are too limited in extent to be of commercial significance, and hence are chiefly of scientific interest.

Extractable Waxes from American Lignites

A comparative study by microscopical observations was made of the melting behavior of waxy products extracted from lignite, of domestic and imported montan wax from lignite, of natural vegetable wax and resin, and of resin hand-picked from coal. The natural waxes tested had low melting points and a narrow temperature range between the initial melting and the free flowing melt. The resins had a relatively high initial melting point and a wide temperature range in complete melting and gave rather viscous melts. Melting of the benzene-extracted products of lignite closely simulated melting behavior of montan wax and natural vegetable wax, whereas the melting characteristics of alcohol-benzene extraction products of lignite indicated a relatively high content of resinous substances. The melting temperature of wax and resin is important in evaluating their commercial usefulness.

Some significant results obtained in the study of the petrography and extractable waxes from American lignites can be summarized as follows:

1. The physical composition of lignitic coals can be determined by microscopic analysis, and a type classification made, based on determined proportions of anthraxylon and attritus present.

2. Attrital lignites tested gave the highest yields of solvent extractable products. Xyloid lignites, that is, those consisting predominantly of anthraxylon, gave very low yields of extractable product.

3. The quality of the extract from attrital lignite can be predetermined to a certain extent by petrographic analysis, since the ingredients in the attritus can be identified with the microscope.

4. The technique of microscopic analysis and type classification should prove to be a useful tool in further consideration of American lignite deposits as domestic sources of extractable waxes.

The megascopic and microscopic descriptions of different varieties of American lignites are given in figure 4, with typical petrographic analyses and solvent extraction yields. Photomicrographs of the lignites as seen in thin section are included in the figure and show the nature of anthraxylon and attritus.

The survey of the wax-extraction yields of American lignites was continued by examination of two lignites from Alabama, four from South Dakota, and one from Texas. Table 2 shows the extraction yields, using benzene and a mixture of 80 percent benzene and 20 percent ethyl alcohol as solvents.

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TABLE 2. - Wax extraction yields, air-dried coal basis

Source of lignite				Wax yields, percent	
State	County	Bed	Mine	Benzene	Benzene-alcohol
Alabama.....	Choctaw	Unidentified	Outcrop	2.8	7.0
Do.	Wilcox	do.	do.	.6	1.6
South Dakota.....	Dewey	Hell Creek	Dewey County	1.2	2.8
Do.	do.	do.	Isabel	1.0	2.2
Do.	do.	do.	Schnabel	1.1	2.7
Do.	Harding	Unidentified	Cave Hills	1.0	2.0
Texas.....	Freestone	do.	Drill Core	1.8	4.1

Methods of Analyzing Coal and Coke

To answer the many requests received by the Bureau regarding the methods used in its laboratories for analyzing coal and coke and for determining various physical and chemical properties of these fuels and their ashes, one of the Bureau's earliest technical papers has been revised and published as a bulletin.7/ First published in 1912 as Technical Paper 8, Methods of Analyzing Coal and Coke, by F. M. Stanton and A. C. Fieldner, it was revised and enlarged in 1913, 1926, 1929 and 1938 to incorporate new methods and improvements in existing methods. The present bulletin includes changes in the method for determination of carbon and hydrogen, methods for determining the agglutinating value and free-swelling index of coal, and tumbler test for coke.

Investigation of Abrasiveness of Coal

Experience in operating pulverized-fuel plants has demonstrated that some coals are much more abrasive than others. Recognizing the need for a method of estimating abrasiveness and in response to numerous requests for information on this important but heretofore unmeasured property of coal, the Bureau has developed and published8/ a tentative procedure believed suitable for trial in estimating coal abrasiveness. The suggested abrasion apparatus is illustrated in figure 5. Essentially, the test consists of rotating four removable wearing blades in a 4-kg. charge of air-dried 4-mesh to 0 coal for a fixed number of revolutions and then determining the loss in weight sustained by the blades. This loss in weight, expressed in milligrams, is considered the abrasiveness of the coal.

Abrasion results with 13 representative coal samples, a sandstone, and a coke are given in table 3.

These samples cover a wide range in rank and ash content. As with any empirical test method, the results are influenced by such variables as speed and duration of rotation, weight and size of coal employed, and clearance between the wearing blades and the walls of the mortar containing the coal. With proper control of these variables, the method gives results that can be reproduced within about 3 percent with very abrasive coals, but with coals of very low abrasiveness, variations up to 18 percent were found. However, this degree of precision probably is sufficient for most purposes.

7/ Fieldner, A. C., and Selvig, W. A., Methods of Analyzing Coal and Coke: Bureau of Mines Bull. 492, 1951, 51 pp.

8/ Yancey, H. F., Geer, M. R., and Price, J. D., An Investigation of the Abrasiveness of Coal and Its Associated Impurities: Min. Eng. vol. 3, March 1951, pp. 262-268.

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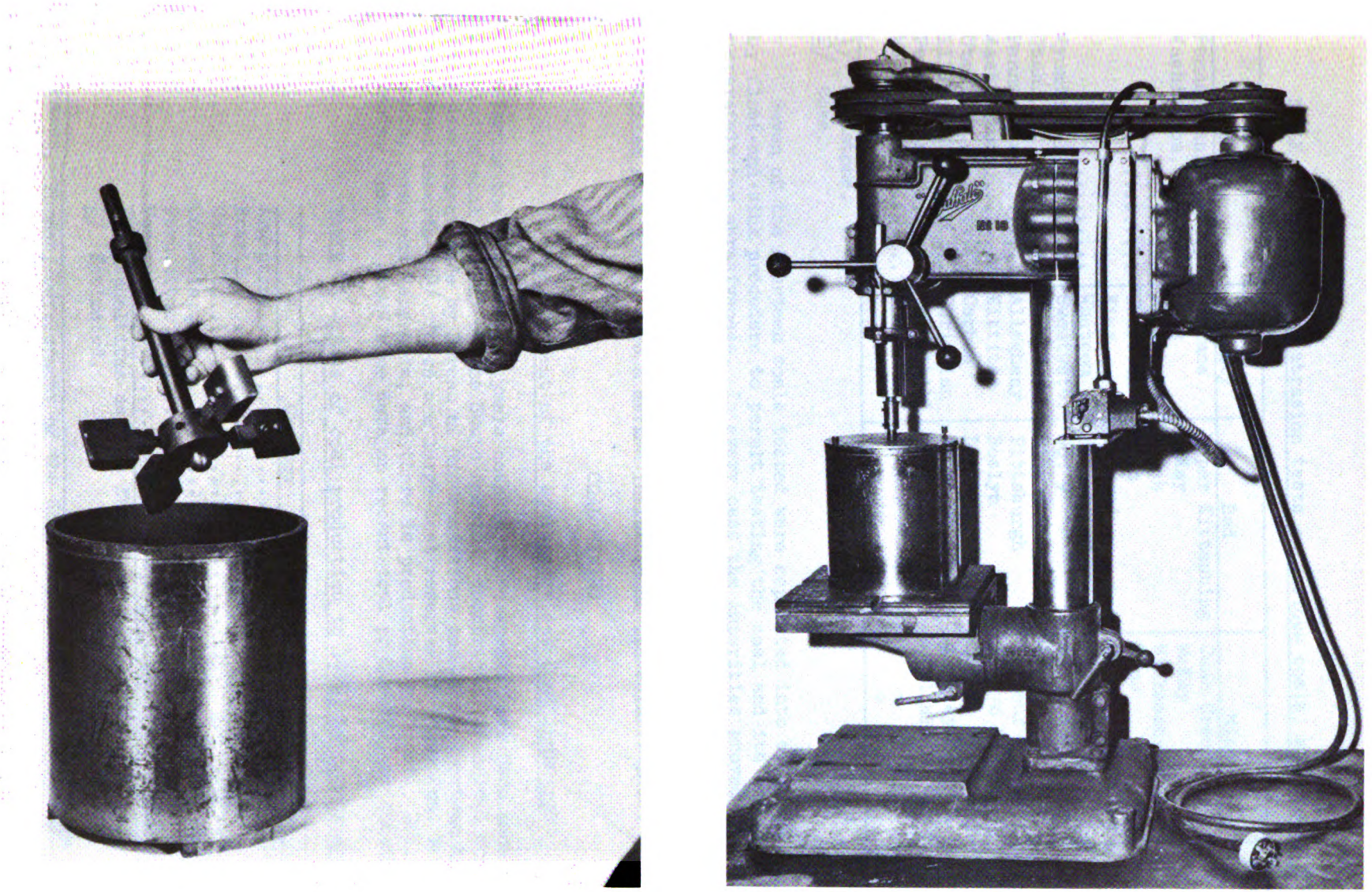

A B

Figure 5. - New apparatus for estimating abrasiveness of coal: A, Mortar, shaft-arm assembly, and wearing blades; B, Mortar and drill press used to drive wearing blades.

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TABLE 3. - Results of abrasion tests on various coals, sandstone, and coke

State	County	Bed	Mine	Abrasion loss, mg.
Pennsylvania.....	Indiana	Upper Kittanning	Cush Creek	12
Washington.....	King	McKay	McKay	18
Do.	Pierce	No. 4	Wilkeson-Wingate	42
Do.	Thurston	Tono	Tono	45
Do.	King	Unnamed	Olson-Cumberland	92
Do.	Whatcom	Bellingham	Bellingham	120
Wyoming.....	Sweetwater	No. 9	D. O. Clark	130
Utah.....	Carbon	Hiawatha	King	165
Pennsylvania.....	Allegheny	Pittsburgh	Montour No. 10	171
Washington.....	Kittitas	Roslyn	Roslyn No. 3	185
Utah.....	Carbon	D	Castle Gate	212
Illinois.....	Christian	No. 6	Langley No. 9	242
Pennsylvania.....	Schuylkill	-	Coaldale	686
Washington[1].....	-	-	-	1,210
Do.[2]	-	-	-	2,506

1/ Sandstone.
2/ Coke.

Seven of the fourteen coals tested were separated into clean coal and impurity by float-and-sink procedure to permit testing the coal and its associated impurities separately for abrasiveness. In every case the impurities proved far more abrasive than the coal substance. Clean coal ranged in abrasiveness from about 10 to 150 milligrams, while the heavy impurities separated from these samples ranged from about 300 to 2,850 milligrams. Thus, the amount and character of the impurities associated with a coal have a marked influence on its abrasiveness.

COAL MINING

Development of the South American Coal Industry

During the past several years notable improvements have been made in the production of coal in many of the South American countries. Technical contributions to this development made by Bureau of Mines missions and other American technicians have been described.9/ There are large coal reserves in Argentina, Brazil, Chile, Colombia, Peru, and Venezuela, and there is production in Brazil, Chile, Peru, and Colombia. Table 4 shows some data on recent coal production, by countries.

TABLE 4. - Rate of coal production in 1948, by countries

Country	Tons
Argentina.....	1/62,000
Brazil.....	2,013,000
Chile.....	2,239,000
Colombia.....	2/1,000,000
Peru.....	187,000
Venezuela.....	21,000

1/ Asphaltites used as fuel.
2/ Estimated.

9/ Fraser, Thomas, Development of the South American Coal Industry: Min. Eng., vol. 190, April 1951, pp. 356-372.

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In Argentina recent developments aimed at expanding the national production of coal comprised: (1) Exploration work in the Rio Turbio coal field of Argentine Patagonia, (2) construction of a narrow-gage railway from that coal field to the Atlantic coast port of Rio Gallegos, and (3) the opening of several exploratory and development mining projects. Some test shipments of cargo size have been made to the industrial plants in Buenos Aires.

In the State of Santa Catarina, Brazil, there has been an extensive development that brought about a production of coking coal capable of supplying half of the needs of the new national steel industry. The new Capivari central preparation plant is shown in figure 6. There are smaller developments in the States of Parana and Sao Paulo. The older industry of the State of Rio Grande do Sul has continued at almost an even rate of production, but modern cleaning plants have been installed to improve the product.

In Chile extensive investigations have led to the use of the central Chilean coals for metallurgical purposes in the new steel industry at Huachipato. In Colombia the estimated annual production of coal has doubled the past several years. There are three producing coal fields, each conveniently located near large industrial centers: One is near Bogota, one near Medellin, and one near Cali. The Bureau of Mines is giving technical assistance, and improvements in mining practice, based on American experience, are being introduced.

In Peru new activity has been centered around the anthracite region of the lower Santa River Valley, which is connected to the Pacific coast port of Chimbote by a meter-gage railway. The three active mines in this region are each capable of producing anthracite at the rate of about 200 tons a day. A modern preparation plant embodying the Chance process and the Hydrotator process has been erected near the port of Chimbote, and shipments of well-prepared anthracite are now being made to South American markets, mainly Argentina, where small proportions of this coal are being used in the utility plants of the city of Buenos Aires.

In Venezuela the coal industry has not been very active because of the ready availability of petroleum. A one-time extensive mining operation of the Venezuelan Government in the Nariqual Valley has not been producing coal for about 5 years. Recently, production of coal was initiated in the western part of the country near the head of Lake Maracaibo, where one new active company has a record of expanding production, although up to the present the total production has not exceeded 100,000 tons a year.

Estimate of Coking-Coal Reserves

The Bureau's investigation of known recoverable reserves of coking coal is being conducted in three parts as given below and as described in a paper presented at a meeting of the American Gas Association and published in the proceedings.10/

1. Estimate of known (measured plus indicated) recoverable reserves of all coking coals.

2. Study of the preparation characteristics of coking coals to determine which are or can be made suitable for metallurgical use by washing or other beneficiating treatment.

10/ Brown, Ralph L., Bureau of Mines Program of Appraising Minable Reserves of Coking Coal: Proc. Am. Gas Assoc., 1940, pp. 483-486.

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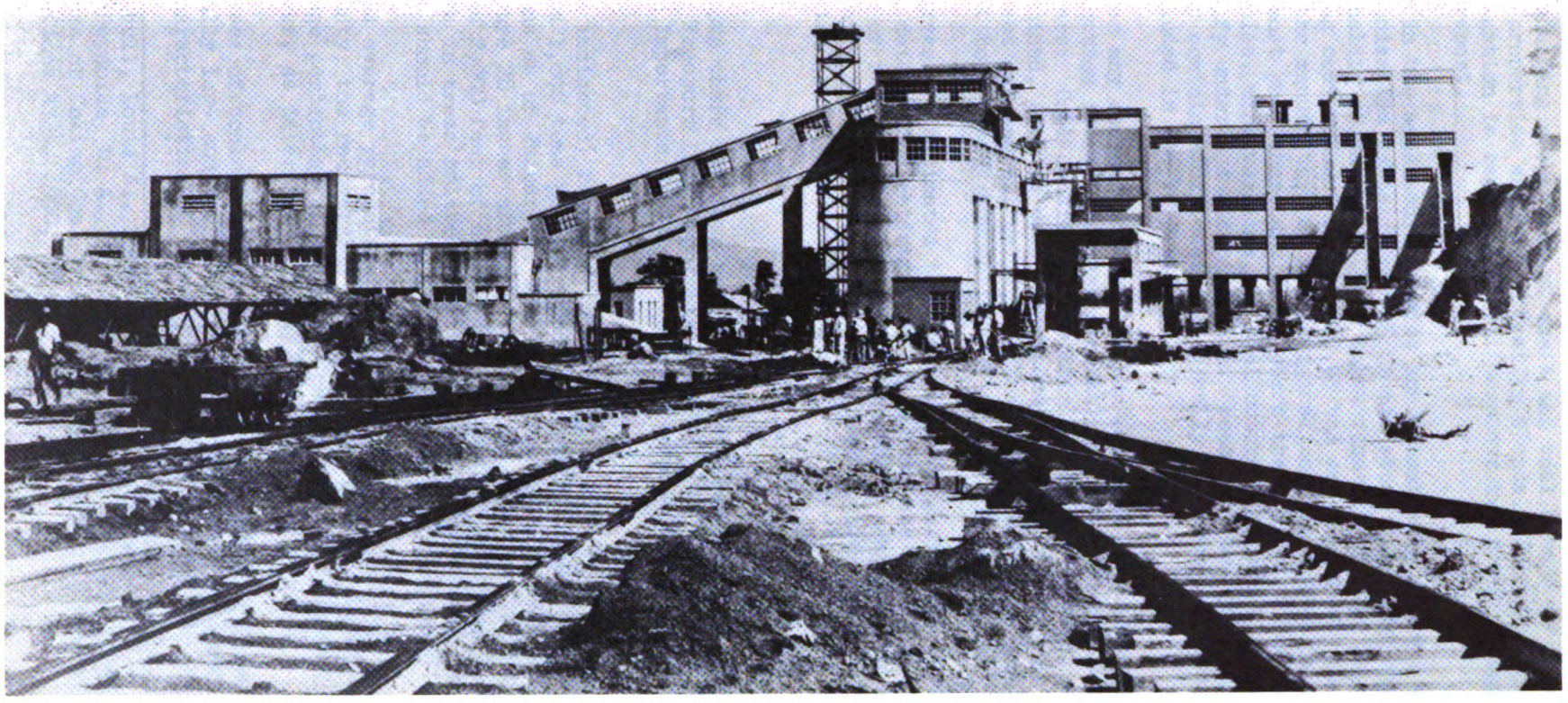

Figure 6. - The Capivarí central preparation plant, Brazil.

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3. Study of the carbonizing properties of coals and coal blends to determine metallurgical coke-making properties and most effective blends. The first part of this comprehensive investigation is reported here, the other two parts at appropriate places later in this report.

All bituminous coals in the Appalachian region are potentially coking, and, therefore, until the carbonization tests in part 3 of the study have been completed to determine the coking quality of the coals, all known reserves of coal in the region are included as coking coal. However, not all of the coals included in these reports are suitable for the manufacture of metallurgical coke according to present-day standards. Also, in most beds there are areas where there are too few (or no) bed sections from drill holes, mine workings, or coal outcrops on which to base estimates that would qualify under the definitions of "measured" or "indicated" reserves. These areas may contain additional geologically inferred reserves, but such reserves are not included in the reports.

All coal remaining for any reason within the mined-out area of a mine is considered a loss. No distinction is made between avoidable or unavoidable losses. Included in these losses are: Coal considered too thin to mine; coal that legally is required to be left unmined, such as coal under some highways, railroads, and rivers; coal left to protect gas and oil wells; and coal left in barrier pillars between mines and adjacent to property boundaries.

The recoverable reserves are estimated tons of unmined coal in beds 28 inches and more thick, as of the date of the estimate, multiplied by the percentage of recovery. The percentage of recovery is the weighted average, computed for each bed, and represents the ratio of tons of coal produced from a given area to the estimated total tons originally in place in that area.

Cambria County, Pa.

The investigation shows there are four major coal beds in Cambria County: Upper Freeport (E), Lower Freeport (D), Upper Kittanning, (C'), and Lower Kittanning (B). Three beds of minor importance are the Middle Kittanning (C), Clarion (A'), and Brookville (A).11/

Known (measured plus indicated) reserves in all beds, based on a minimum bed thickness of 14 inches and 1,800 tons per acre-foot of coal in place, are estimated at 2,407 million short tons as of January 1, 1948. Of this total, 1,909 million tons is in beds that measure 28 inches and more thick. All reserves in Cambria County are under less than 2,000 feet of overburden. If future drilling or development should prove reserves in areas omitted from the estimate because of insufficient data, such reserves should be added to the total estimated reserves.

Based on the weighted average percentage of recovery for all beds 28 inches and more thick in Cambria County, the recoverable reserves are estimated at 931 million tons. The weighted average percentage of recovery for all beds in Cambria County, including all mining losses, as determined by this investigation is 48.77.

Representative analyses of coals from the four major beds show that Cambria County coals are principally low- and medium-volatile bituminous. A summary of the coking and preparation characteristics of these coals indicates that they are coking

11/ Dowd, James J., Turnbull, Louis A., Toenges, Albert L., Cooper, H. M., Abernethy, R. F., Reynolds, D. A., and Fraser, Thomas, Estimate of Known Recoverable Reserves of Coking Coal in Cambria County, Pa.: Bureau of Mines Rept. of Investigations 4734, 1950, 25 pp.

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but, in some areas, do not meet present metallurgical standards as to ash and sulfur content. The Upper Freeport, Lower Freeport, Upper Kittanning, and Lower Kittanning coals have been important sources of supply for the manufacture of metallurgical coke for many years, but are usually used in blends with other coals. The Kittanning coals in Cambria County respond well to washing for sulfur reduction, but the Freeport coals are more difficult to wash.

Indiana County, Pa.

The investigation shows there are three major coal beds in Indiana County: Upper Freeport (E), Lower Freeport (D), and Lower Kittanning (B). Five beds of minor importance are the Pittsburgh, Upper Kittanning (C'), Middle Kittanning (C), Clarion (A'), and Brookville (A).12/

Known (measured plus indicated) reserves in all beds, based on a minimum bed thickness of 14 inches and on 1,800 tons per acre-foot of coal in place, are estimated at 2,544 million short tons as of January 1, 1948. Of this total, 1,989 million tons is in beds 28 inches and more thick. All coal beds for which reserves have been estimated in Indiana County are under less than 2,000 feet of overburden. As in Cambria County, areas were omitted where insufficient data were available, and reserves in these areas, if later proved by drilling or development, should be added to the total.

Based on the weighted average percentage of recovery of 48.95 percent for all beds in Indiana County, the recoverable reserves in beds 28 inches and more thick are estimated at 974 million short tons.

Representative analyses of coal from the major producing beds show that the coals of Indiana county are principally medium- and high-volatile A bituminous rank. In summarizing their carbonization and preparation characteristics, the report indicates that these coals generally coke strongly and may be used either singly or as blends to make metallurgical coke, when sufficiently low in ash and sulfur. The Upper and Lower Freeport beds usually contain two or more shale or bone partings, and in many locations in the county the beds are high in sulfur. However, much of the sulfur is present as thin streaks and flakes of iron sulfides that can be removed by mechanical cleaning, and in some cases a high-grade metallurgical-coking coal and a lower-grade steam coal may be produced by a three-product separation in the preparation plant.

Pike County, Ky.

Results of the study of coking-coal reserves in Pike County, Ky.,13/ are given in the third report in this series. There are four coal beds of major importance in Pike County: Upper Elkhorn No. 3, Upper Elkhorn No. 2, Upper Elkhorn No. 1, and Lower Elkhorn. Beds of secondary importance are the Winifrede, Williamson, and Bingham. Ten other beds, in addition to numerous uncorrelated lenses, are of minor importance.

12/ Dowd, James J., Turnbull, Louis A., Toenges, Albert L., Cooper, H. M., Abernethy, R. F., Reynolds, D. A., and Crentz, William L., Estimate of Known Recoverable Reserves of Coking Coal in Indiana County, Pa.: Bureau of Mines Rept. of Investigations 4757, 1950, 22 pp.

13/ Dowd, James J., Turnbull, Louis A., Toenges, Albert L., Abernethy, R. F., and Reynolds, D. A., Estimate of Known Recoverable Reserves of Coking Coal in Pike County, Ky.: Bureau of Mines Rept. of Investigations 4792, 1951, 34 pp.

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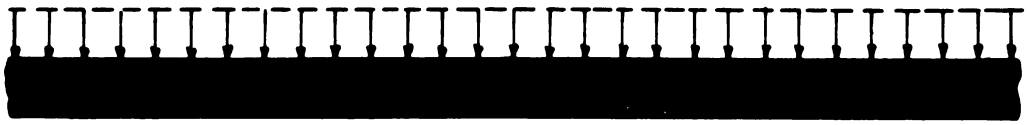
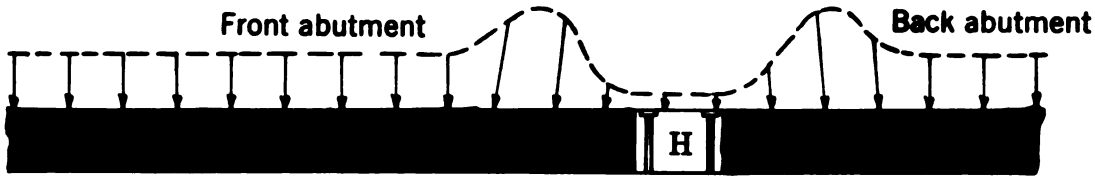
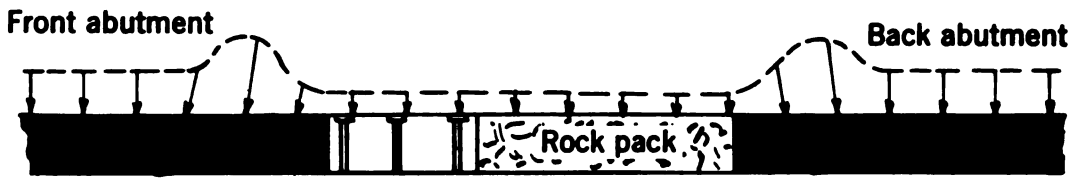


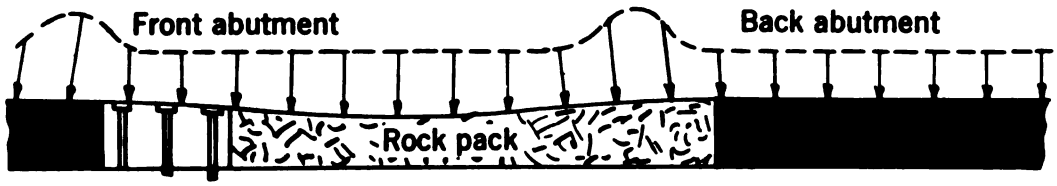
A-Virgin coal: Uniform load over entire seam



B-Opening a new face: Load increased at abutments



C-Coal face advancing: Front abutment advancing



D-First weight: Back abutment load transferred to pack

Figure 7. - Distribution of roof load in vicinity of coal face.

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Known (measured plus indicated) reserves in all beds based on a minimum bed thickness of 14 inches and 1,800 tons per acre-foot of coal in place are estimated at 3,916 million short tons as of January 1, 1948. Of this total, 3,189 million tons is in beds 28 inches and more thick. All known reserves in Pike County are under less than 2,000 feet of overburden. These totals do not include reserves in areas omitted because of insufficient data. Based on the weighted average percentage of recovery of 55.08 percent for all beds 28 inches and more thick in Pike county, the recoverable reserves are estimated at 1,757 million tons.

Analyses of representative samples show that the coals of Pike county are high-volatile A bituminous, and that the moisture, ash and sulfur contents usually are low. A summary of the carbonizing properties indicates that highly fissured coke generally is obtained from eastern Kentucky coals when carbonized singly; however, when blended with coal of higher rank and carbonized, a strong coke is produced.

Mining Methods and Practices

Anthracite Mechanical Mining Research

The effects of pressures in mining at depth and methods for controlling these pressures in mines of the United Kingdom were studied.14/

Figure 7 shows distribution of roof load in the vicinity of the coal face, as determined by measuring the loads borne by packs in the United Kingdom. Drawing a lesson from the successful experience of the mining engineers of the United Kingdom, it can be said that the only practical solution of roof-control problems that now prevail in the Pennsylvania anthracite region, which will become increasingly important with future mining at greater depths, lies in a more technical and scientific approach than has been used generally in this country. Instruments must be provided and techniques developed for determining the forces that prevail so that these forces may be controlled intelligently and economically.

To develop instrumentation for measuring loads borne by roadway supports, the Bureau of Mines arranged for the design and construction of a continuous recorder and load cells. As a beginning, a set of 12 cells was installed on collars of conventional 3-piece wood sets in the coal gangway of a steeply pitching bed of the Southern anthracite field for the purpose of recording loads and changes in loads as mining progressed. After several months of use, these instruments were removed for incorporating improvements.

In 1947 a lightweight universal shearing machine of German origin was provided and installed by the Bureau of Mines in a steeply pitching bed of the Southern anthracite field for test purposes. These cutting tests represent the first use of a machine of this kind in steep-pitch mining in the United States.15/

The machine is driven by compressed-air and weighs 3,000 pounds. It is equipped with a 7-foot bar and a 33-block chain, both of conventional design. The machine is rated at 20 hp. by the manufacturer, and this output was obtained with air at 80 pounds per square inch pressure. At 60 p.s.i. the output was found to be between 16

14/ Buch, John W., and Allan, Andrew, Jr., Some Roof-Control Practices in Coal Mines of the United Kingdom: Bureau of Mines Inf. Circ. 7599, 1951, 7 pp.

15/ Buch, John W., and Allan, Andrew, Jr., Anthracite Mechanical Mining Investigations. Progress Report 3. Preliminary Testing of Korfmann Universal Shearing Machine, Model SK 20: Bureau of Mines Rept. of Investigations 4794, 1951, 11 pp.

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and 17 hp. Tests made in anthracite of medium hardness showed that an output of less than 6 hp. is required for cutting with this machine. Cuts 8 feet high and 6 feet deep were made in less than 10 minutes, with air pressure of 50 p.s.i. and air consumption at the rate of less than 400 cubic feet per minute.

The apparent ease with which this machine cuts anthracite may be attributed to its hand feed in contrast to power feed of conventional American machines; to the compressed-air motor, which cannot be overfed without stalling and thus operates with a light-touch ripping effect on brittle coal; and to the "widia" (tungsten-carbide) tipped cutter bits, which remain sharp longer than some other types. Originally designed to meet conditions in mines of the German Ruhr, it is not to be expected that the machine will meet Pennsylvania anthracite conditions fully without some modification. However, the tests showed that the rate of driving gangways in steeply pitching anthracite beds can, by its use, be increased sufficiently to justify further tests and development.

Bituminous-Coal Mining Studies

Studies of the use of mechanical mining equipment for extraction of pillars were continued in two mines in West Virginia and three in eastern Kentucky, and comparative data were obtained from mines not extracting pillars. Arrangements were made for testing an imported German coal planer at a specially developed longwall face in a relatively thin bed of Pocahontas No. 3 coal in Raleigh County, W. Va., and part of the equipment was received from Germany. A 25-year analysis of haulage equipment used in bituminous-coal mines16/ showed that belt-conveyor haulage expanded rapidly in the last 5 years, that there has been a substantial growth of rope haulage in recent years, and that the number of locomotives in use remained substantially constant, with gradual change in types over the years studied.

Studies on the Use of Diesel Engines Underground

Besides the well-known products of combustion in the exhaust gas from Diesel engines, there are many minor constituents that escape detection by ordinary methods of analysis because of their low concentration. Mass spectrometric analysis (table 5) has shown a number of such trace constituents that reveal the extreme complexity of the chemical reactions that occur during combustion.17/ The presence of olefins, for example, is evidence of thermal decomposition, and the appearance of oxygenated hydrocarbons indicates direct oxidation.

The concentration of carbon monoxide and of aldehydes in the exhaust gas of a 2-stroke-cycle, 3-cylinder Diesel engine were studied as a function of fuel-to-air ratio and of engine speed. A minimum of 0.03 to 0.06 volume-percent of carbon monoxide was found at a fuel-to-air ratio of about 0.015 to 0.020. The concentration of carbon monoxide did not vary with engine speed over the range of fuel-to-air ratios studied. The minimum concentration of aldehydes, on the other hand, was about fifty times as high (5 p.p.m.) at 1,300 and 2,000 r.p.m. as it was at 600 r.p.m. (0.1 p.p.m.).

16/ Young, W. H., and Anderson, R. L., Underground-Haulage Trends at Bituminous Mines: Coal Age, vol. 56, No. 6, June 1951, pp. 96-97.

17/ Elliott, M. A., Davis, R. F., and Friedel, R. A., Products of Combustion from Diesel Fuel: Proc. Third World Petrol. Conf., sec. VII, Preprint 25, June 1951, 17 pp.

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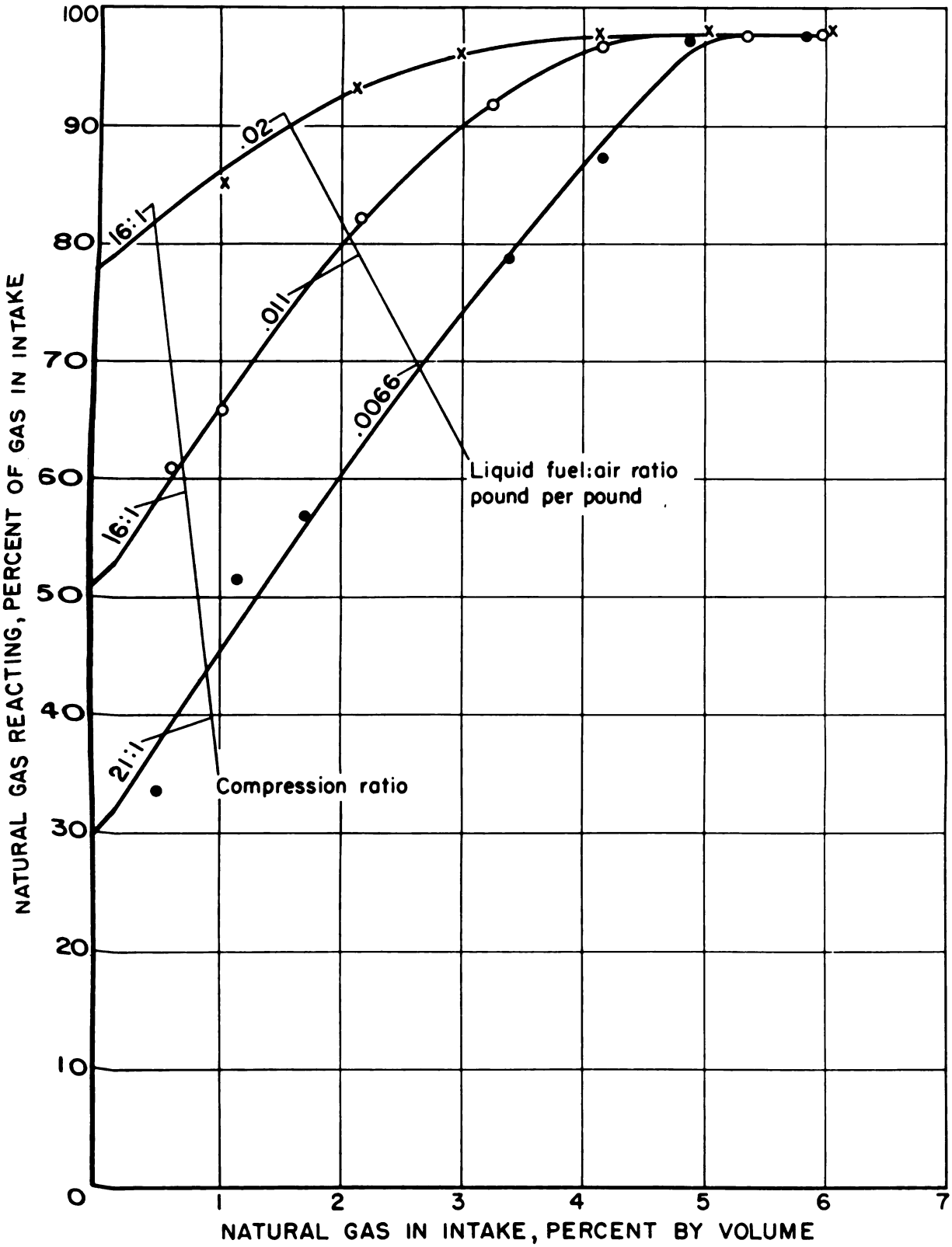

Figure 8. - Relation between proportion of natural gas reacting, concentration of gas in intake, and liquid fuel-air ratio in tests of a CFR Diesel engine.

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TABLE 5. - Mass spectrometer analysis of exhaust gas from a 6-cylinder, 4-stroke-cycle Diesel engine1/

Haldane gas analysis		Mass spectrometer analysis		Mass spectrometer analysis	
Product of combustion usually determined	Percent by volume	Hydrocarbons	P.p.m. by volume	Oxygenated compounds	P.p.m. by volume
CO_2.....	1.68	Methane.....	4.5	Formaldehyde.....	98.8
CO.....	.10	Ethylene.....	16.2	Acetaldehyde.....	31.5
O_2.....	18.50	Propylene.....	5.3	Methanol.....	5.7
C_nH_{2n+2}.....	.00	Butylene.....	6.6	Acetic acid.....	49.3
N_2.....	79.72	Pentylene.....	10.4	Propionic acid....	7.4
		Hexylene.....	10.5	Acetone.....	17.8
		Acetylene.....	1.0 (?)	NO.....	29.0
		Butadiene.....	.5 (?)	NO_2.....	.004
		Benzene.....	Trace		
		Toluene.....	Trace		
		Other hydrocarbons2/	50		

1/ Engine operating conditions: Speed, 675 r.p.m,; load, none; fuel consumption, 2.52 lb. per hr.; fuel-air ratio, 0.0085 lb. per lb.

2/ In addition to those listed, includes paraffin hydrocarbons.

The operation of dual-fuel Diesel engines is of increasing economic importance, especially in areas where there is low-cost natural gas. Dual-fuel combustion means the simultaneous burning of liquid and gaseous fuel. The gaseous fuel is generally mixed uniformly with the intake air. The liquid fuel is injected into the compressed gas-air mixture and furnishes a source of ignition for the mixture.18/ Figure 8 shows the relation between the proportion of natural gas reacting, concentration of gas in intake, and the liquid fuel-to-air ratio in a test of a 4-stroke cycle CFR Diesel engine. Substantially all of the gas reacts, regardless of liquid fuel:air ratio, when the concentration of gas reaches the lower limit of flammability. Under the test conditons this is about 5 volume percent. If the concentration of combustible gas is greater than the lower limit of flammability, the flame will propagate throughout the gas-air mixture when the liquid fuel ignites. The attendant rapid rate of heat release may result in engine-operating difficulties and "knock." If the concentration of gas is below the lower limit of flammability, the gas is not burned completely. The presence of unreacted gas in the exhaust indicates that high temperature regions do not permeate the entire combustion space. The fraction of gas reacting increases with an increase in liquid fuel-air ratio, with concentration of combustible gas in the gas-air mixture, and is affected by type of engine and by engine speed. The lower limit of flammability of natural gas was 5 percent; propane, 1.9 percent; butane, 0.9 percent; and hydrogen, 10 percent.

Three commercial Diesel mine locomotives were tested to determine if they met the requirements of Schedule 22.19/ Two of these were designed and built in the United States, and one in England. Tests disclosed design features in each that required modification, and these modifications, which were worked out with the manufacturers on the basis of the permissibility tests, have been or are being made.

18/ Elliott, M. A., and Davis, R. F., Dual-Fuel Combustion in Diesel Engines: Ind. Eng. Chem., vol. 43, 1951, (in press); Am. Chem. Soc., Div. Petrol. Chem. and Div. Gas and Fuel Chem., Joint Symposium on Combustion Chemistry, April 1951, pp. 267-290.

19/ Procedure for Testing Diesel Mine Locomotives for Permissibility and Recommendations on the Use of Diesel Locomotives Underground: Bureau of Mines Schedule 22, 1944, 31 pp.

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Control of Coal-Mine Fires

At a cost of less than 1 cent a ton, more than 120,000,000 tons of bituminous and anthracite coal have been saved from fire and destruction as the result of the Bureau of Mines program of controlling fires in inactive coal deposits throughout the United States during the past 3 years. At current market prices, it is estimated that this coal would be worth over $500,000,000 when mined. Since Congress authorized the fire-control work in the 1949 fiscal year, 21 projects have been undertaken by the Bureau of Mines, and 15 of them are now completed. In addition to saving the coal, health hazards from noxious gases released by these fires have been eliminated, and many millions of dollars worth of private and public property have been saved from possible damage or destruction.

In widely scattered areas of the country, some of the fires in inactive coal deposits had been burning for a generation or more. Fires on public domain were put out or controlled entirely at the expense of the Federal Government. The Government provided two-thirds of the funds for controlling fires on private, State, and county lands, with the landowners supplying one-third of the cost. Various methods have been used in fighting mine fires, including sealing, flooding, use of carbon dioxide, and combinations of these methods. Sealing was used to control an outcrop fire in a steeply pitching bed near Rifle, Colo.[20/] Although the 21 fires in inactive coal deposits, which have been or are being controlled by the Bureau of Mines, include some of the worst ones known to exist, Bureau officials have investigated more than 80 uncontrolled fires of this type in different parts of the country.

Coal-Mine Explosions and Fires

Improvement is evident in the reduction of explosion and fire hazards in coal mines, as shown by the records for 1949.[21/] The safeguards and attention accorded to these hazards since 1946 apparently have reduced the loss of life and damage to property, although the number of fires and ignitions has not been decreased. Deaths from explosions and fires in 1949 totaled 17, compared with 48 in 1948 and an average of 106 for the years 1944-47.

Loss of life in coal-mine explosions in 1949 was 8, compared with 46 in 1948 and an average of 78 in the 5 years from 1944 to 1948. The total of 8 deaths was the smallest since 1870, the first year for which records were kept. A total of 14 coal-mine explosions was studied in 1949, of which 6 were in anthracite mines. This compares with an average of about 25 explosions for the preceding 5 years and with 29 explosions reported in 1948.

In 1949, 30 coal-mine fires were reported, with 4 deaths, compared with 16 fires and 7 fatalities in 1948 and an average of 18 underground coal-mine fires annually for the 5-year period 1944-48.

Causes of Fatalities in Mines

The investigation of major and minor coal-mine disasters has been a regular function of the Bureau of Mines since its creation in 1910, and the facts obtained

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20/ East, J. H., Jr., Russell, H. W., and Bolmer, R. L., How a Coal-Outcrop Fire was Controlled: Min. Cong. Jour., vol. 36, No. 1, January 1950, pp. 24-25.

21/ Forbes, J. J., Fene, W. J., and Humphrey, H. B., Coal-Mine Explosions and Coal- and Metal-Mine Fires in the United States in 1949: Bureau of Mines Inf. Circ. 7572, 1950, 17 pp.

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during such investigations have been used as a basis for recommendations made to forestall similar occurrences. A comprehensive study of individual fatal coal-mine accidents was not attempted until February 10, 1950, when the policy of investigating all coal-mine fatalities in the United States was approved. These investigations are now being conducted by Federal coal-mine inspectors functioning under the provisions of the Coal-Mine Inspection and Investigation Act of May 1941, Public Law 49. Reports covering the investigations are distributed to management, the State mine-inspection agency, and representatives of the mine workers' organization having jurisdiction at the mine.

A study covering the Federal investigations of 76 of the 92 fatalities that were charged to the anthracite industry of Pennsylvania during the calendar year 1950[22] revealed that 64 percent of these fatalities were charged to falls of roof, face, or rib; 15 percent were charged to haulage, almost half of them occurred on the surface; and the remainder was from miscellaneous causes. It was determined that approximately 71 percent of these fatalities were the result of human failure.

A study was made of 263 of the 315 fatalities from falls of roof, face, or rib charged to the bituminous-coal industry during the calendar year 1950.[23] In 1950, 67 percent of all fatalities that occurred underground at bituminous-coal mines resulted from falls of roof, face, or rib. Three of every four of these accidents occurred within 25 feet of the working face. Again, three out of four fatalities in this zone occurred in the area from the last permanent roof support and the face. Only one of the fatalities resulted from a fall of roof that was bolted. The data show that roof bolting is an effective means of reducing roof-fall accidents. The chief factors responsible for the roof-fall fatalities were found to be: Failure by the face boss or foreman to give adequate supervision, negligence by the employee, reliance on roof testing as a substitute for roof supports, and acceptance of the hazard as not meriting stronger protective measures.

A study of 131 fatal haulage accidents from 14 coal-mining States and Alaska was another part of the program to reduce the hazards mainly responsible for mine fatalities.[24] Records of the past year show that haulage accidents caused 27 percent of the coal-mine fatalities, while haulage personnel represented only 12 to 15 percent of the mine employees. Of the 121 fatal injuries investigated, 102 occurred underground, 14 on the surface, and 5 at strip operations. The most frequent types of accidents were: Falling or stumbling into the path of moving equipment, caught between equipment and roof or timber, derailments, and collisions. Physical hazards were: Scant clearance, inadequate retarding devices, and defective equipment. Unsafe practices were: Excessive speed, disregard of rules or instructions, riding in unsafe position, pushed trips, getting on and off moving equipment, and negligent operation.

Toxic Mine Atmospheres

The Bureau of Mines has developed an approval system for respiratory protective devices[25] as part of its continuing work in setting forth the minimum requirements

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22/ Forbes, J. J., and Weaver, H. F., Why Men were Killed at Pennsylvania Anthracite Mines in 1950: Bureau of Mines Inf. Circ. 7609, 1951, 13 pp.

23/ Forbes, J. J., Back, T. L., and Weaver, H. F., Falls of Roof: The No. 1 Killer in Bituminous-Coal Mines: Bureau of Mines Inf. Circ. 7605, 1951, 11 pp.

24/ Ankeny, M. J., and Kingery, D. S., Analysis of Haulage Fatalities in Bituminous-Coal Mines in 1950: Bureau of Mines Inf. Circ. 7604, 1951, 28 pp.

25/ Pearce, S. J., Bureau of Mines Approval System for Respiratory Protective Devices: Bureau of Mines Inf. Circ. 7600, 1951, 6 pp.

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that various types of equipment should meet to be considered safe and satisfactory for use in certain hazardous or unhealthful conditions. Provisions have been made for testing and approving the following types of respiratory protective devices: Self-contained breathing apparatus, supplied-air respirators, gas masks, dispersoid respirators, and chemical cartridge respirators. These devices are tested at the Central Experiment Station of the Bureau of Mines in Pittsburgh, Pa., self-contained breathing apparatus by the Safety Branch and the other types of respirators by the Health Branch.

Bureau of Mines approval of respiratory protective devices is based upon performances tests rather than upon specifications as to how the devices must be built. The submission of respiratory protective devices to the Bureau of Mines for approval is entirely voluntary on the part of the manufacturer, the Bureau having no regulatory power requiring that all respirators be submitted for approval testing. The Bureau acts merely as an impartial testing agency that makes available to the public a list of respiratory protective devices that have met definite performance requirements, but wide acceptance of Bureau of Mines approval of these devices encourages the manufacturers to submit them for approval. The Bureau of Mines checks the approved respirators that are on the market from time to time to ascertain if they conform to the device originally approved in physical make-up and in performance.

In 1950, 9 new approvals and 34 extensions of approval were granted on respiratory protective equipment, including industrial gas masks, supplied-air respirators, and respirators for protection against particulate matter. Numerous check tests were made on approved protective equipment purchased in the open market, and any deficiencies disclosed were brought to the attention of the manufacturers.

In connection with the work carried out under the provisions of the Federal Coal-Mine Inspection Act, approximately 14,000 samples of mine air were analyzed to determine the adequacy of ventilation in coal mines, to detect and aid in the elimination of hazards from flammable and toxic gases in mines, and to aid in the control and extinguishment of mine fires.

Surveys conducted in coal mines to determine the concentrations of air-borne dust produced by mining operations, such as drilling rock strata above and below the coal measures, were directed toward providing information for the formulation of recommendations for control measures to reduce exposure of coal-mine workers to dusts that are potentially harmful, because they tend to cause silicosis, a lung disease. As a result of cooperative tests of dust-collecting devices, which were being developed commercially, several collectors were developed that are capable of reducing to acceptable limits the dust disseminated into the air of working places during roof drilling for roof bolting.

More than 1,600 examinations were conducted to determine the concentration and particle size of air-borne dusts, and the composition of such dusts and dust-source materials, with particular reference to free silica content, to evaluate their hygienic significance in the working environment of coal and other types of mines. These examinations were conducted by microscopic, petrographic, X-ray diffraction, and spectrographic methods. Examinations were made also of various materials to determine their suitability for use in rock-dusting coal mines to reduce the hazard of coal-dust explosions.

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Electrical Equipment for Mines

Tests of equipment for approval as "permissible" for use in gassy mines were conducted during the year.26/ Types of equipment submitted for testing approved in 1950 included air compressors, drills, loading and conveying machines, mining machines, trucks for moving machines, pumps, locomotives, power trucks, distribution boxes, timbering machines, cleaners, sprayers, flashlights, and multiple-shot blasting units.

There is need for better methods and improved protective devices in coal mines, because electrical hazards from fires and explosions resulting from electrical failures are greater in the coal-mining industry than in any other industry. Studies and tests designed to determine the possible value of the short-circuited contactor as an electrical protective device for coal-mine service were investigated.27/ Preliminary tests were made to determine the operating characteristics of the device that is intended primarily for protection against damage from accidental short circuit, after which tests were made under actual service in three coal mines.

The mine operator who uses electrical equipment has, in general, the choice of installing fuses or circuit breakers to protect his equipment from the effects of electrical failures. In many instances both devices are used in the same mine. Although fuses and circuit breakers have been used widely for many years, they have objectionable characteristics that are inherent in their designs, which have not been overcome after many years of service experience in coal mines. It is common knowledge that a rubber-insulated cable, such as used with mobile equipment in mining service, can be badly damaged or completely ruined by a short circuit in the cable when a fuse of proper size to permit starting current is provided at the cable nip. This is due to the fact that a fuse has considerable time lag before "blowing" and the resistance of the arc produced by the short circuit in the cable has a limiting effect upon the current. As a result, the arc may be maintained with a current value below that required to melt the fuse element, and the cable may be badly damaged before the circuit is deenergized and the arc extinguished.

The tests of this investigation and information from other sources indicate that the short-circuiting contactor offers possibilities for the development of a simple, rugged, inexpensive protective device for mine electrical circuits. Such a contactor, in combination with an ordinary fuse, can be made to provide high-speed, short-circuit protection for electrical circuits approaching that of the best air circuit breakers.

Roof Control

Although the principles of supporting mine roofs remain the same, changes in mining roof-support methods make it necessary to relate new practices to the fundamental principles and to revise earlier handbooks issued on these subjects. As a part of the program to reduce the fatalities and injuries from roof falls, a concise explanation in question and answer form has been compiled for use as a text in educational courses, which are conducted by the Bureau for miners.28/ The text covers the stresses leading to roof failure, the methods of putting in timbers, roof bolts, or other forms of support, and safe practices that should be followed. Sketches and photographs illustrate most of the points discussed. The education accomplished with the aid of this text is essential to the reduction of roof-fall accidents.

26/ Brunot, H. B., Permissible Mine Equipment Approved During the Calendar Year 1950: Bureau of Mines Inf. Circ. 7606, 1951, 8 pp.

27/ Harrison, L. H., The Short-Circuiting Contactor as an Electrical Protective Device for Coal-Mine Sercice: Bureau of Mines Rept. of Investigations, 4759, 1951, 11 pp.

28/ Forbes, J. J., Thomas, Edward, and Barry, A. J., Questions and Answers on Roof Support in Bituminous-Coal Mines: Bureau of Mines Handbook, 1951, 90 pp.

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Original installations of roof bolts in American mines have been found in widely separated mining districts. A few of the known installations have been in place as long as 40 years. A study of the history[29] of the practice in the United States shows that some early attempts were not continued, because the expense was above that of conventional timbering and was not compensated for by the added efficiency of mechanical mining.

Although considerable theoretical information has been published on the subject of roof control, little knowledge is available that can be applied practically by the mining engineer. The Bureau of Mines has equipped a roof-control laboratory at College Park, Md., and a crew at work in the field with "stratascope" will provice basic engineering data to supply this need. Preliminary experiments show considerable promise.

Anthracite Flood Prevention

In continuing the Bureau of Mines work on the underground mine-water problem in the anthracite region of Pennsylvania, data from a study on the design, installation, and operation of centrifugal pumps used in the region were published[30] as possible assistance for mines in which pumping problems are met.

A clay, sand, and gravel deposit known as the "Buried Valley" of the Susquehanna River in the Northern Field of the anthracite region makes great care necessary in mining operations to avoid breaking the strata between the mine workings and these valley-fill deposits to avoid inundation that would result from such break-through. Operators of mines in this area must be familiar with the water-bearing deposits and surrounding strata to conduct their operations safely. A study was completed of the mine-water problem concerned with the "Buried Valley."[31] Engineers of the Bureau of Mines have ascertained the accuracy of mine plans and maps, the size and thickness of barrier pillars necessary to hold water safely in different circumstances, the effects of faults, the porosity of barriers and of strata, the method of approach to abandoned or flooded mine workings, the use of bore holes, the precautions to be observed in and after tapping water, and the building of dams in the mines.

PREPARATION OF COAL

Coal Washing

International Conference on Coal Preparation

The First International Conference on Coal Preparation was held in Paris in June 1950. The coal-preparation staff of the Bureau of Mines collaborated with the appropriate divisions of the National Coal Board of France and the British National Coal Board in organizing this Conference, and the Director of the Bureau of Mines served on the committee for selecting papers to be presented. Cerchar, the research organization of the French Coal Board, was host to the meeting. Twelve papers were prepared and presented by American authors, three of them by members of the Bureau staff.

29/ Thomas, Edward, Roof Bolting in the United States: Bureau of Mines Inf. Circ. 7583, 1950, 8 pp.

30/ Lesser, William H., Centrifugal Pump Installations in Anthracite Mines of Pennsylvania: Bureau of Mines Rept. of Investigations 4749, 1950, 22 pp.

31/ Ash, S. H., Buried Valley of the Susquehanna River: Anthracite Region of Pennsylvania: Bureau of Mines Bull. 494, 1950, 27 pp.

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One of these papers described several methods of examining coal-washing-plant operations and preparing charts and formulas for expressing the efficiency of performance for purposes of comparison.32/ Another paper described the experimental development of the kerosine-flotation process for handling fine coal and the performance of some commercial installations that have been made in the Birmingham field, Ala.33/ The third paper described an improved method of estimating particle size in subsieve size particles.34/ This process was developed by the Heyl and Patterson Co., incidental to a cooperative agreement between that company and the Bureau of Mines.

In the committee meetings at which the Bureau was represented, it was tentatively planned to set up an international committee for standardizing coal-preparation test procedure and formulas for appraising preparation-plant performance.

Evaluating Coal-Washery Performance

The Bureau of Mines has made available to the American coal industry the ideas on the evaluation of coal-washery performance, developed at the International Conference on Coal Preparation, which seem most important from an American viewpoint.35/ The various methods of evaluating washery performance are illustrated with previously published data and with some new data obtained as the result of experimental work undertaken since the conference to provide information on certain controversial points.

Performance can be evaluated in a number of different ways, with the choice of the proper method to use being dictated by the objectives of the investigation and the data available. The performance of any cleaning unit is determined by the character of the raw coal treated, the density of the separation, and the sharpness of the separation effected between the coal and impurity. Thus, three factors enter into performance; one is determined by the coal, one is dictated by market or use considerations, and one is an inherent characteristic of the cleaning unit itself. Efficiency, which is the ratio of the actual to the perfect separation, is influenced by all three factors. It measures directly the loss of saleable coal in the refuse and thus is the performance criterion of greatest practical value to the coal-preparation plant operator.

Sharpness of separation or sharpness of cut between coal and impurity is measured in terms of either error area or probable error, both of which are determined from a distribution curve for the washing operation. These criteris are useful in

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32/ Yancey, H. F., and Geer, M. R., [A Critical Examination of Methods Used in Evaluating Coal-Washery Performance] : Rev. ind. minérale, Special Issue No. 1, November 1950, pp. 4-12. Pres. at International Conference on Coal Preparation, Paris, France, June 1950.

33/ Gandrud, B. W., [Kerosine Flotation: A Process for Cleaning and Dewatering Fine Sizes of Coal] : Rev. ind. minérale, Special Issue No. 2, December 1950, pp. 217-243. Pres. at International Conference on Coal Preparation, Paris, France, June 1950.

34/ Notary, Joseph A., Gorman, Henry B., and Fraser, Thomas, [Test Method for Appraising the Performance of Sludge Recovery and Water-Clarifying Equipment] : Rev. ind. minérale, Special Issue No. 2, Dec. 1950, pp. 171-179. Pres. at International Conference on Coal Preparation, Paris, France, June 1950.

35/ Yancey, H. F , Geer, M. R. Efficiency and Sharpness of Separation in Evaluating Coal-Washery Performance: Min. Eng., vol. 3, June 1951, pp. 507-517.

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comparing the operation of washing units making dissimilar separations, because they are substantially independent of either the character of the raw coal or the density of separation. They must not be confused with efficiency, however, because low values of error area or probable error sometimes are accompanied by low efficiency. Table 6 gives a comparison of washery-performance factors for 26 individual washing tests in terms of density of separation, efficiency, percentage of misplaced material, error area, and probable error. These data demonstrate clearly that there is no direct correlation between efficiency and misplaced material on the one hand and error area and probable error on the other. A sharp separation is an efficient one when made on a coal of favorable washability characteristics, but even a very sharp separation may result in a relatively low efficiency if made at a low density or if the coal contains an abnormal amount of material of intermediate density.

TABLE 6. - Comparison of performance criteria for a number of washing operations

Cleaning unit	Size of coal, inches or mesh	Specific gravity of separation[1]	±0.10[2]	Error area	Probable error	Percent: Misplaced material	Percent: Efficiency[3]
Heavy medium.....	Over 1/4	1.56	7.0	6	0.011	1.4	-
Do[4].....	1-1/4 to 3/8	1.50	4.8	24	.031	.7	99.9
Do[4].....	2 to 1/4	1.57	22.5	28	.040	5.8	97.1
Do.....	1-5/8 to 15/16	1.79	20.3	48	.074	3.5	-
Do[4],[5].....	14 to 35	1.63	1.7	24	.044	.4	99.9
Do[4],[5].....	8 to 200	1.65	10.4	28	.027	3.0	98.5
Jig, Baum.....	5 to 0	1.57	7.0	63	.087	4.8	97.5
Do.....	5 to 0	1.46	14.4	68	.111	21.0	72.2
Do.....	3 to 0	1.67	10.0	76	.086	8.7	96.1
Do.....	3 to 0	1.53	5.8	81	.103	3.4	98.0
Jig, pulsator.....	3 to 1-1/4	1.45	19.8	57	.057	7.9	94.0
Do.....	1-1/4 to 1/4	1.70	2.3	95	.130	2.2	99.6
Jig, plunger.....	1-1/4 to 0	1.63	4.3	78	.088	6.0	97.3
Jig, basket.....	1 to 0	1.72	4.9	93	.130	4.5	97.8
Wet table[4].....	3/32 to 0	1.78	6.7	63	.075	4.4	98.0
Do[4].....	1/4 to 0	1.70	1.9	79	.103	2.5	99.3
Rheolaveur.....	4 to 5/16	1.63	10.4	75	.096	8.5	98.8
Do.....	5/16 to 0	1.70	13.5	130	.294	7.1	94.4
Menzies cone.....	3 to 2	1.55	8.7	31	.035	2.9	99.3
Do.....	3/4 to 3/8	1.65	3.8	63	.078	2.5	98.2
Hydrotator.....	11/16 to 28	1.83	4.2	63	.099	2.6	99.0
Froth flotation...	10 to 0	1.57	3.6	73	.127	4.3	98.2
Do.....	10 to 0	1.69	4.7	125	.234	7.2	96.4
Humphreys spiral[4]	8 to 0	2.04	-	147	.367	-	91.8
Pneumatic table[4].	1/2 to 1/4	1.68	2.0	94	.131	3.2	98.3
Contraflow.....	2 to 0	1.79	1.8	98	.179	3.0	99.2

1/ The specific gravity at which the distribution curve crosses the 50 percent ordinate, indicating equal distribution of material between washed coal and refuse.
2/ Not adjusted for material heavier than 2.0 specific gravity.
3/ Fraser and Yancey formula: yield of washed coal divided by yield of float coal of same ash content.
4/ Laboratory test, all others plant tests.
5/ Cyclone.

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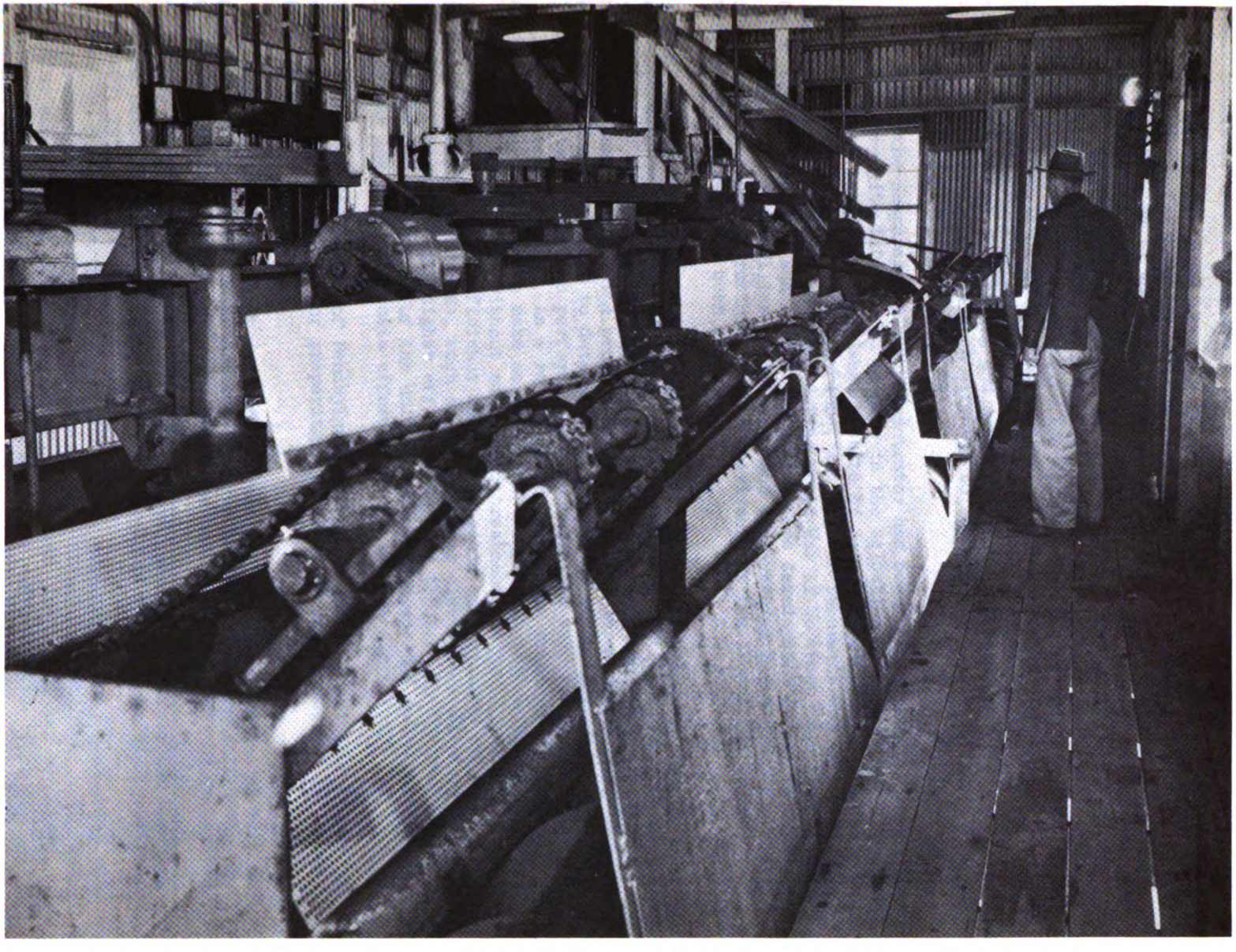

Figure 9. - Kerosine-flotation cells in an Alabama coal-preparation plant.

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Cleaning and Recovery of Fine Coal

Another commercial application of the kerosine-flotation process, developed by the Bureau of Mines for cleaning and dewatering fine sizes of coal, makes three such plants now in operation in the Birmingham, Ala., district. (See fig. 9.) One of these plants treats an underflow product from dewatering screens; the other two treat fine coal (approximately 14-mesh to 0) wet-screened out of the run-of-mine coal. The fines that are screened out of the run-of-mine coal are removed, cleaned, and dewatered directly, thus preventing the formation of sludge and slurry and the problems always associated with them. Another advantage of the process is that fine sizes of high-grade coking coal formerly wasted are now recovered. The latest developments in this method of cleaning and dewatering fine-size coal have been published.[36/] Subsequent investigations have been directed to improvement in the design of the flotation cells to increase their capacity.

Recently, the Humphreys spiral concentrator has been investigated[37/] to provide the coal industry with information to help in the selection of coal-cleaning equipment and to supplement previously published results of detailed performance studies of the wet table, pneumatic table, Dutch cyclone, and jigs of pneumatic, plunger, pulsator, and Baum types. The spiral was used during World War II to treat the chrome-bearing sands of Coos Bay, Oreg., and in the years since its development it has been adopted for treating ores, nonmetallics, and coal. One coal-washing plant using the spiral concentrator went into operation in the Pennsylvania anthracite fields in 1946, and another plant has recently been completed.

Coals from Alabama, Kentucky, West Virginia, and Washington were tested in the spiral concentrator, all samples representing natural 8-mesh fines screened from run-of-mine coal except the West Virginia coal, which was prepared by crushing 3-inch slack to pass 8-mesh. The products from the spiral were examined by float-and-sink and screen-sizing tests to determine the mechanics of the separation between coal and impurity. The basic performance characteristics were found to be the same for all of the coals tested. As a result of the modifying influence of particle size, the spiral is unable to make an efficient two-product separation between coal and impurity on these coals without additional treatment. Also, a classified feed can be treated with higher efficiency than is possible on a natural raw coal; consequently, semiclassified feeds, such as silt-bank or classifier-underflow materials, can probably be treated with higher efficiencies than those shown for raw coals. The inherently low efficiency of the unit may be overcompensated under certain conditions by the low operating costs afforded by its extreme simplicity.

In the anthracite region of Pennsylvania, a field study of existing commercial launder-screen installations for preparing fine sizes of anthracite was initiated in September 1950.

To supplement this field study, a full-scale pilot launder screen has been designed and constructed at the Anthracite Research Laboratory, Schuylkill Haven, Pa. This unit is undergoing tests at a regional fine-coal cleaning plant in cooperation

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36/ Gandrud, B. W., and Riley, H. L., Recent Developments in Combination Cleaning and Dewatering of Fine Sizes of Coal: Bureau of Mines Rept. of Investigation 4707, 1950, 28 pp.

37/ Geer, M. R., Yancey, H. F., Allyn, C. L., and Eckhouse, R. H., Laboratory Performance Tests of the Humphreys Spiral as a Cleaner of Fine Coal: Am. Inst. Min. and Met. Eng. Tech. Pub. 2944F, Trans, Am. Inst. Min. and Met. Eng., vol. 187, 1950, pp. 1057-1067; Min. Eng., vol. 187, October 1950, pp. 1057-1067; discussion, Trans. Am. Inst. Min. and Met. Eng., vol. 187, 1950, pp. 1160-1162; Min. Eng., vol. 187, November 1950, pp. 1160-1162.

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with the Hudson Coal Co., Scranton, Pa. This pilot unit is 18 feet long by 1 foot wide and is faced with 38-mesh stainless-steel cloth of approximately 60 percent open area. It is being used to determine the maximum quantity of fine coal that can be recovered from material formerly lost in the plant effluent.

Preparation Characteristics of Coking Coal

Cambria County, Pa.

In the preparation phase of the Bureau's investigation of known recoverable coking-coal reserves, the first three reports in a planned series, giving in detail the preparation characteristics, by counties, of the principal coal beds in each county, were published. The first report, covering Cambria County, Pa.,[38] shows that the main washing problem, as in the other two counties studied, concerns the release of sulfur. The important coal reserves are in the Upper Freeport, Lower Freeport, Upper Kittanning, and Lower Kittanning beds. Crushing the Kittanning coals to fine size gave a significant sulfur reduction in the float coal. The Freeport beds, particularly the Upper bed, were not markedly responsive to crushing for release of sulfur. All samples collected in Cambria County, except one Lower Freeport-bed coal sample, indicated that a satisfactory metallurgical-grade washed product could be obtained by mechanical cleaning alone or mechanical cleaning in conjunction with fine crushing. The quality of the Lower Freeport-bed coal tends to improve toward the northern part of the county.

Indiana County, Pa.

According to the second report, which covers Indiana County, Pa.,[39] the principal coal reserves are found in the Upper Freeport, Lower Freeport, and lower Kittanning beds. Isolated patches of Pittsburgh-bed coal are present, and the Upper Kittanning bed is found in minable thickness at some locations, but production from these sources is not large. The Lower Kittanning bed is moderately responsive to fine crushing for release of sulfur impurities, while the effect of crushing upon the release of sulfur from the Upper and Lower Freeport beds at many places is significant from a practical standpoint. Although the coal from all three important beds at certain selected locations shows a low sulfur content, the upgrading of large areas of coal in Indiana County to metallurgical standards is a difficult technologic undertaking. Upper Freeport-bed coals containing 1.75 percent or less sulfur can be upgraded to metallurgical standards without difficulty but raw coals containing more than 1.75 percent sulfur usually require precise control over the washing operation and economic disposal of a high-ash middling product. The quality of the Lower Kittanning bed tends to improve toward the northern part of the county, while the reverse appears to be true in the case of the Lower Freeport bed.

Armstrong County, Pa.

The important coal deposits in Indiana County extend to the west into Armstrong County.[40] In addition to a small output from the Pittsburgh and Upper Kittanning

38/ Crentz, William L., and Steele, Fern, Preparation Characteristics of Coal Occurring in Cambria County, Pa.: Bureau of Mines Rept. of Investigations 4747, 1950, 40 pp.

39/ Crentz, William L., Steele, Fern, and Bailey, A. L., Preparation Characteristics of Coal Occurring in Indiana County, Pa.: Bureau of Mines Rept. of Investigations 4763, 1951, 33 pp.

40/ Crentz, William L., Steele, Fern, and Bailey, A. L., Preparation Characteristics of Coal Occurring in Armstrong County, Pa.: Bureau of Mines Rept. of Investigations 4788, 1951, 25 pp.

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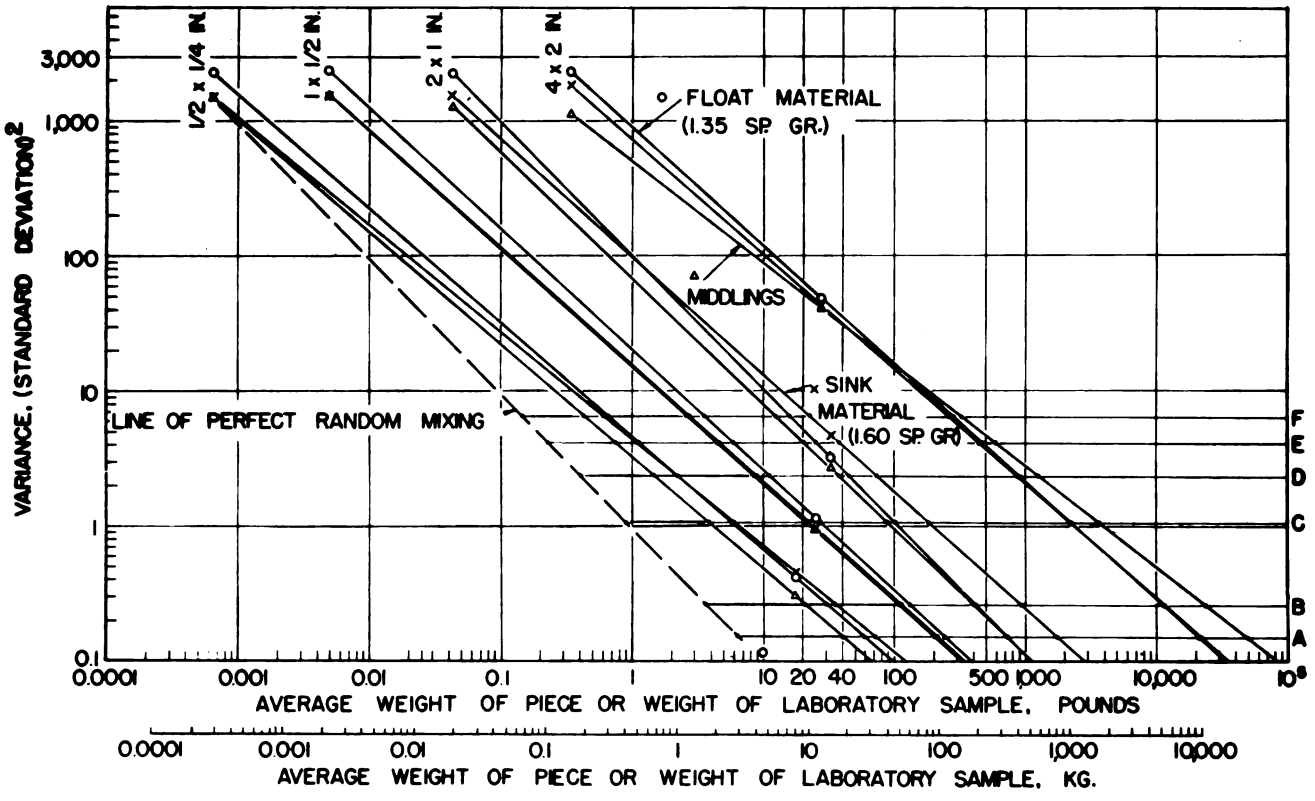

Figure 10. - Float-and-sink sampling characteristics of four sizes of a western Pennsylvania bituminous coal.

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beds, the Middle Kittanning bed is mined to a small degree in Armstrong County. The Lower Freeport bed is opened at some places, but it is usually too thin and spotty in occurrences to support large-scale operations. A study of the washing characteristics of the Armstrong County coals shows that the Upper Freeport and Lower Freeport beds are responsive to fine crushing for release of sulfur impurities, whereas the Lower Kittanning bed is unaffected. The Upper Freeport bed is the major source of production and offers interesting possibilities as a source of coking coal. The sulfur in the coal ranges from less than 1 percent to more than 3 percent without forming a perceptible geographic pattern. Precise preparatory methods would upgrade the Upper Freeport bed to metallurgical standards over a wide area. Conclusive examination of the washing characteristics of the Lower Freeport bed in Armstrong County is hampered by the small number of mining operations. The meager data obtained indicates a possibility of preparing a satisfactory metallurgical fuel from the coal in the northwest section of the county. The Lower Kittanning bed, which is secondary in importance to the Upper Freeport bed, is a high-ash and high-sulfur deposit. The high sulfur content, much of which occurs in those forms of sulfur that cannot be removed by specific-gravity separation, removes this bed from consideration as a source of metallurgical coal.

Float-and-Sink Control Tests

An intensive study[41] of coal sampling and float-and-sink testing was made to provide statistical data for estimating the size of samples needed for precise work in float-and-sink testing. The basic data obtained in these tests covered the size range of 4 inches to 1/4 inch, divided for testing into four size increments (that is, 4- by 2-inch, 2- by 1-inch, 1- by 1/2-inch, 1/2- by 1/4-inch). By statistical analysis of the data obtained in these experiments, the fundamental curves of figure 10 were projected to show the relation between particle size or sample weight and standard deviation in test results. Based on these curves the estimates of table 7 were set up to show the sample required in relation to size of particles in the coal sample.

TABLE 7. - Weights of laboratory sample required to fulfill accuracy of lines A to F, figure 10, for degree of mixing obtained and for perfect random mixing, in pounds

Line	Float		Middlings		Sink	
	As found	Perfect mixing	As found	Perfect mixing	As found	Perfect mixing
Size: 1/2 by 1/4 in.						
A	59.0	11.0	40.0	7.0	72.0	7.0
B	32.0	6.0	21.0	4.0	36.0	4.0
C	5.8	1.5	3.8	.9	5.9	1.0
D	2.5	.7	1.5	.5	2.3	.5
E	1.2	.4	.8	.3	1.1	.3
F	.7	.3	.5	.2	.6	.2
Size: 4 by 2 in.						
A	21,500	5,600	49,500	2,700	22,000	4,400
B	11,500	3,200	24,000	1,550	11,500	2,500
C	2,260	780	3,650	375	2,200	600
D	910	350	1,250	170	860	270
E	480	196	580	96	440	154
F	285	125	320	61	255	97

41/ Landry, Bertrand and Bailey, Arthur L., Coal Sampling for Float-and-Sink Control Tests: Rev. ind. minérale, Special Issue No. 2, December 1950, pp. 123-133. Pres. at International Conference on Coal Preparation, Paris, France, June 1950.

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Research Pilot Plant

A coal-preparation research pilot plant with facilities for experimental treatment of coal on a pilot-plant scale has been erected on the Experimental mine property of the Bureau of Mines at Bruceton, Pa.[42] This research plant is in the same building with the boiler plant serving the Synthetic Liquid Fuels Research and Development laboratories, and the equipment is arranged so that a common coal-handling and refuse-disposal facility serves both plants. The pilot plant consists of a permanent installation of coal-handling equipment to unload coal from railway cars or trucks, convey it into the building, and distribute it to a series of overhead bins adapted to feed any desired preparation facilities that may be installed in the pilot-plant area. The pilot-plant area now has a 5-foot heavy-medium separating cone, with the necessary accessory equipment to make a complete heavy-medium washing unit; a complete ball mill pulverizing plant, with provisions for drying coal in the mill; and an installation of froth-flotation cells adapted for treatment of test lots of fine coal by the froth-flotation process, or for the reconditioning of nonmagnetic-flotation media for the heavy-medium washing system.

Coal Preparation for Synthetic Fuels

The Bureau has investigated the bituminous-coal fields of the United States containing large reserves of bituminous or subbituminous coals that may be economically used for synthesis of liquid fuels.[43] Samples were taken in coal fields of Pennsylvania, Ohio, West Virginia, Indiana, Illinois, Kentucky, Utah, Wyoming, Montana, and North Dakota. These were tested to show how the coal would be expected to respond to washing treatment for preparing a fuel adapted to synthetic fuels manufacture. The coals examined varied greatly in their responsiveness to preparatory treatment. While major emphasis was placed on ash reduction in these tests, other characteristics, such as petrographic constituents and sulfur content, were affected by preparatory treatment in a measure usually somewhat comparable to the ash reduction. These factors, as well as preparation losses and operating costs, must all be considered in appraising the value of washing these coals.

Drying Low-Rank Coals

Fluidized Drying

As part of the general investigation of processing and upgrading low-rank coals, experimental work conducted to date in the fluidized drying pilot plant has given further proof that the fluidized drying process is amenable to advance calculation as to rates and yield, and that any noncoking fuel can be dried by this technique. On the basis of previously reported work and a report summarizing 14 tests made on subbituminous coal from Colorado, Wyoming, and Alaska, and lignites from Texas and Greece,[44] the following conclusions were made:

42/ Fraser, Thomas, and Barrett, O. T., Federal Bureau Operates Coal-Preparation Research Plant: Mechanization, September 1950, pp. 111-119.

43/ Fraser, Thomas, Crentz, William L., and Barrett, Orrin T., Preparation Characteristics of Some Coals Available for the Synthetic Liquid Fuels Industry: Bureau of Mines Bull. 495, 1950, 132 pp.

44/ Parry, V. F., and Wagner, E. O., Drying Fine Coal in the Entrained and Fluidized State: Min. Eng., vol. 187, September 1950, pp. 974-982. Trans. Am. Inst. Min. and Met. Eng., vol. 187, 1950, pp. 974-982.

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1. The capacity of a drying unit is about 500 pounds of water liberated by the fuel per hour and square foot of cross-sectional area.

2. It is practical to remove 90 to 93 percent of the total moisture in the coal by drying in the fluidized column without apparent devolatilization or modification of the chemical structure of the coal.

3. The rapid evolution of moisture and shrinkage of the coal cause considerable degradation of the coal particles during drying. In general, the decrease in average sizes of the coal particles is approximately equal to the bed moisture of the coal treated.

4. The drying of a given coal to any given degree is a straightforward heat balance problem.

5. The dried coal dusts are free flowing and flow through orifices by gravity at rates similar to the flow of fluids. Tests show that the flow of dust in pounds per minute equals $14.5 \times D^{2.65}$, where D is the diameter of the orifice in inches.

6. It is estimated that the investment costs of fluidized drying units should be in the range of $150 to $200 per ton of daily capacity, depending on the moisture content of the coal, and the costs of removing water from coal in the fluidized dryer should range from $0.85 to $1.00 per ton of water removed, based on cost data for 1948.

Modified Fleissner Process

In a study of the mechanism of drying lignite in a modified Fleissner[45/] process using steam at 400 p.s.i.g., it was indicated that the degree of drying and disintegration is determined by: (1) Size of lignite; (2) time of contact between lignite and drying agent; (3) temperature of drying agent; (4) humidity of drying agent; and (5) porosity of lignite bed in drier. The steam consumption for drying was calculated as 0.8 pound per pound of water removed; final moisture content of dried lignite was approximately 14 percent; and the lump size of lignite was preserved much better than in other methods of drying.

Storage of Lignite

Lignite recovered during the construction of the Garrison dam across the Missouri River, north of Bismarck, N. Dak., is being placed in storage in large million-ton piles. During the last year, 380,000 tons of lignite was stored, bringing the total in storage to 880,000 tons. When the Garrison Dam is completed, approximately 3 million tons of lignite will have been recovered and stored. The Bureau's method of stockpiling, worked out successfully on several piles of Colorado subbituminous coals, has to date been successful with this North Dakota lignite, when proper methods of piling were used. Under the conditions prevailing at the Garrison Dam site, the investigations indicate that this lignite can be stored for indefinite periods in large uncapped piles without firing spontaneously, if the Bureau's methods of piling are rigorously followed.

As part of an informal service to lignite consumers on the storage of lignite, many industrial stockpiles have been visited, and advice has been given on methods

45/ Oppelt, W. H., The Mechanism of Drying Lignite: Proc. North Dakota Acad. Sci., vol. 4, 1950, November, 1950, pp. 33-39.

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of safely storing lignite. Also, experimental work on storage of steam-dried lignite has been continued in a simulated silo-type storage of dried minus-3/8-inch lignite. During a 15-month period of observation, no signs of hot spots or spontaneous ignition were noted, indicating that this type of storage is suitable for smaller sizes of steam-dried lignite.

UTILIZATION OF COAL AND OTHER FUELS FOR COMBUSTION

Fuel-Engineering Service

Fuel-engineering service to Government establishments in the selection, testing, and use of fuel and fuel-burning equipment and in the economical use of steam was continued. Services on 195 special problems were given 40 different Federal agencies, in addition to those described in the following paragraphs.

At the request of the Veterans' Administration total boiler-plant operating-cost comparisons were made of the use of oil, gas, and coal, and the proper type of fuel-burning equipment was recommended for four projects. A study was made of a hospital boiler plant at Dwight, Ill., and recommendations were made for new boilers, new fuel-burning equipment, and fuel; recommendations were made on the proper choice of fuel-burning equipment for a large boiler plant at Perry Point, Md., for new fuel-burning equipment at Augusta, Ga., and for a new project at Manila, Philippines.

A series of tests was also made for the Veterans' Administration on chain grate-type fuel-burning equipment, normally burning rice, to determine the relative performance of rice and barley anthracites of various size consists and volatile contents.46/ Efficiency and capacity were limited by the grate-link design, as well as the design of the furnace, when barley was substituted for rice. Careful maintenance and operation were particularly necessary with barley. In general, the efficiency and capacity decreased with decreased fuel size and volatile content, the ranges on the 250-hp. boiler tested being from 63 to 74 percent in efficiency and from 130 to 170 percent in boiler rating. With barley, the size consist affected both efficiency and capacity to a greater extent than the volatile content, but with a normal amount of undersize the efficiency decrease from the substitution of barley for rice was about 3 percent.

Fuel-cost comparisons were made for three projects for the Federal Power Commission and for one project for the Department of Agriculture. Acceptance tests as to efficiency and capacity of new boiler equipment at the Bryant Street pumping station were made at the request of the Department of Engineering of the District of Columbia. A survey and recommendations were made for hot water and heating equipment for two buildings at the Bureau of Mines Mount Weather Station, Bluemont, W. Va. A new domestic type of automatic stoker for burning anthracite was tested at the request of several Federal agencies. Cooperative work with the Air Preheater Corp. of Wellsville, N. Y., on the prevention of deposits and corrosion on air preheaters was continued.

At the request of the General Services Administration, many operating tests were made on experimental auxiliary air-jet equipment installed to prevent smoke emission from large, underfeed stoker-fired boilers, when using cheaper high-volatile coal, and to improve efficiency at the higher loads. These tests were part of a comprehensive program to determine the best equipment for several plants of this type being operated in the Washington area. General Services administration was also supplied

46/ Burdick, L. R., and Morgan, R. E., Burning Anthracite Barley on a Chain-Grate Stoker in a Two-Arch Furnace: Bureau of Mines Rept. of Investigations 4720, 1950, 7 pp.

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with information and recommendations on the decentralization of coal purchasing and the development of methods of purchasing coal in various regions.

Performance tests of specially twisted metal strips inserted in the tubes of fire-tube boilers to improve heat transfer were made at the request of the Department of the Navy. They operate a great number of boilers of this type, and it was found that an average fuel saving of about $130 per year per boiler could be expected from such equipment.

At the request of the Public Buildings Administration and the Federal Supply Service, plans were developed for a new coal-storage supply of about 100,000 tons for the Washington area, the general type of coal decided upon, the specification analytical limits determined, and recommendations as to award made. Federal coal-purchasing agents were supplied needed information on mines for use in making awards of contracts, the bids for supplying coal to a number of agencies were studied, and recommendations made for award.

An investigation was made of the possible increased use of wood fuel in case of the need for greater defense efforts. A paper[47/] on the burning of wood waste for commercial heat and power was published covering the composition and burning characteristics of wood, the types of wood-waste fuels, their heating values and analyses, the transportation and storage of wood, furnace designs for wood burning, and a tabulation of general information on present-day wood-burning installations.

A handbook[48/] was prepared from basic information on the use of propane and butane fuels, previously published as Bureau of Mines information Circular 7519. The handbook covers the properties of these fuels, the safety precautions to be observed in their use, the methods of transportation, the various purposes for which the fuels are used, the equipment needed, and the amounts used in the United States.

Boiler Feed-Water Conditioning

Analyses and resulting recommendations were made on 9,714 samples of boiler water during the fiscal year as follows: 3,582 from the Department of the Army; 1,908 from the Department of the Air Force; 1,701 from the Veterans' Administration; 663 from the Department of the Navy; 547 from the Post Office Department; 446 from the District of Columbia Government; 293 from the Public Housing Administration; 225 from the Department of Justice; 184 from the Bureau of Indian Affairs; 64 from the Federal Security Agency; 32 from the General Services Administration; 21 from the Public Health Service; 16 from the Department of Commerce; 13 from the Department of Agriculture; 11 from the Bureau of Mines; 4 from the Smithsonian Institution; 3 from the Bureau of Reclamation; and 1 from Howard University. This is about a 4-percent increase over the previous year.

Reports and recommendations covering 27 analyses of various scales, sludges, and deposits, 22 miscellaneous special analyses, and 5 analyses of boiler compounds were made.

A total of 1,148 special Bureau of Mines field water-test kits, 12,764 bottles of chemical reagents, and 14,781 test-kit replacement items was distributed to

47/ Barkley, J. F., and Morgan, R. E., Burning Wood Waste for Commercial Heat and Power: Bureau of Mines Inf. Circ. 7580, 1950, 12 pp.

48/ Barkley, J. F., Questions and Answers on Propane and Butane Fuels, Question and Answer Handbook 4, 1950, 28 pp.

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various Government activities. These amounts are increases over the previous year of about 98 percent, 24 percent, and 23 percent, respectively. Distributing these kits and reagents from a central source saved Government agencies about $20,000 this year as compared to open market purchase.

Consulting service was given to the Red Lake Indian Mills, Redby, Minn., on the use of chemicals bought on specification to replace a more costly proprietary boiler compound; the Bureau of Yards and Docks, Department of the Navy, on the use of boiler water antifoams and on an operating manual for treatment of boiler water in the Marianas area; the St. Elizabeth's Hospital, Washington, D. C., on the control of brine corrosion in refrigeration plant; the Forest Products Laboratory, Department of Agriculture, Madison, Wis., on the control of sludge formation; the Office of the Chief of Engineers, Department of the Army, on chemical analyses procedures for use in Europe; the District of Columbia Government on the use of external feed-water treating equipment and on the chemical treatment of hot-water heating systems; the District of Columbia filtration plant on boiler-water treatment for low-pressure boilers; the Public Housing Administration on boiler corrosion at Cambridge, Mass.; the Department of the Air Force on condensate-line corrosion in stations in Alaska; the Veterans' Administration on control of deposits in nozzles in spray equipment and on return-line corrosion; and the Housing Authority of Baltimore on setting up a central boiler-water control and research laboratory.

At the request of the Potomac River Naval Command, Washington, D. C., visits were made to their local laboratory to instruct in boiler-water testing; to the Marine Barracks, Washington, D. C., where recommendations were made on control of steam losses due to leaking traps and on control of boiler-scale formation; to the Marine Barracks, Quantico, Va., on return-line corrosion problems; to the Naval Communications Station, Washington, D. C , and the Naval Air Station, Anacostia, D. C., on boiler-water difficulties; and to the Naval Research Laboratory, Anacostia, D. C., on making boiler-water tests, on design of chemical and feeding equipment, and on the control of chemical dosages.

Various agencies requested that studies be made at their installations as follows: Boiler plant of the Public Housing Administration, Beltsville, Md., where recommendations were made on boiler-water treatment, resulting in appreciable savings from the elimination of a proprietary boiler-water compound; Naval Hospital, Bethesda, Md., where a comprehensive study of return-line corrosion was made and the causes determined; Naval Powder Factory, Department of the Navy, Indian Head, Md., where the cause of excessive boiler-water carry-over and methods for its prevention were determined and methods were recommended for the elimination of excessive return-line corrosion costing about $20,000 annually; Naval Hospital, Bureau of Yards and Docks, Department of the Navy, St. Albans, Long Island, N. Y., where tests and studies were made to verify the need for a hydrogen-zeolite softener about to be purchased for control of return-line corrosion, but it was found that satisfactory water conditions could be maintained without the need for this equipment, resulting in a saving of an expenditure of about $10,000; Navy Photographic Center Laboratory of the Naval Air Station, Anacostia, D. C., where recommendations were made for cleaning sediment from the water filters and for cleaning slime in the film-rinsing tanks and cheaper chemicals bought on specifications were substituted for a proprietary compound for treating refrigeration water; Detention Headquarters, United States Department of Justice, New York, N. Y., where the boiler plant was to be surveyed, recommendations were to be made on boiler-water treatment, and the engineer was to be instructed in boiler-water testing and control of chemical treatment.

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A Bureau of Mines handbook on boiler-water treatment[49] covers essential information needed by operators, particularly in Government power plants, for applying and controlling chemical treatment of boiler water. The handbook includes chapters on boiler-water causticity and how it is maintained in control; a description of different types of chemical feeders and methods of adjusting dosages; a discussion of the use of soda ash, caustic soda, and phosphate chemicals for control of boiler-scale formation; instructions on the use of tannin in boiler-water treatment; a discussion of control of corrosion in boilers during operating and idle periods and on the use of volatile amines for control of corrosion in return lines; instructions on boiler-water blow-down and its control; and an explanation of boiler-water analyses and how to apply such information to the control of chemical residuals in boiler water.

Boiler-Water Research

Research on the fundamental causes of corrosion in steam condensate-return lines included studies of preventative chemicals, such as amines and ammonia, particularly from the standpoint of lowering the costs of necessary chemical treatment. Contrary to some impressions in industry that the use of amines for this purpose is quite expensive, a written discussion of a paper[50] emphasizing the idea of high costs of amine treatment pointed out that field tests have shown a cost of less than $300 for an entire year for three large housing developments of 4,227 dwelling units, or about 75 cents per million pounds of steam. In other examples cited, the average yearly costs ranged from $57 to $150. In these cases, it was found that the amine morpholine proved to be the most economical amine to use.

At the request of the Department of Justice, an investigation was made of supposed difficulties in dyeing from the use of an amine, where steam was used in direct contact with wool material. It was found that the difficulties resulted from the presence of iron rust previously accumulated in the pipelines.

A laboratory study of a method of removing corrosion products that plugged condensate-return piping in a Government-operated heating plant showed there was some merit in using polyphosphates for this purpose, but a saving of about $1,000 per year resulted when equally active material available on the open market was substituted for a proprietary compound previously used.

A summary on the results of about 2,000 tests obtained with 600 embrittlement detectors in 500 stationary boiler plants and of approximately 500 tests on 20 locomotive boilers was presented at the 1950 Annual Meeting of the American Society of Mechanical Engineers.[51] The results showed the effectiveness of sodium nitrate, quebracho tannin, and zero-caustic treatments to prevent embrittlement cracking. Nitrate and quebracho are inhibitors against intercrystalline corrosion in hot, concentrated caustic solutions. The zero-caustic treatment prevents the formation of concentrated solutions of caustic that cause corrosion. The survey also showed that the frequency of test-specimen failure was not affected by the A.S.M.E.-recommended sulfate-to-carbonate ratios, since it was approximately the same as when no

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49/ Goldman, Louis, Boiler-Water-Treatment Manual for Federal Plant Operators: Bureau of Mines Handbook 5, 1951, 94 pp.

50/ Berk, A. A., Discussion of "New Polar Film Treatment for the Control of Return-Line Corrosion." by H. L. Kahler and J. R. Brown: Proc. Tenth Annual Water Conf., Eng. Soc. of Western Pennsylvania, 1949, pp. 121-122.

51/ Berk, A. A., The Prevention of Embrittlement Cracking: Combustion, vol. 22, No. 6, December 1950, pp. 52-53.

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treatment was used. The report also discussed the 26 cases of cracking of stationary boilers studied during the period 1942-49. Of these, 24 were definitely due to embrittlement cracking, and cracks were found in butt straps, drum metals, tube seats, and tube ligaments. The boiler drums were welded in six of the plants and were riveted in the others. Embrittlement-detector tests did not show enough nitrate or a tannin-type inhibitor to be present in concentrations to prevent cracking, although five of the plants were maintaining the prescribed sulfate to carbonate ratios at the time of failure. However, the effectiveness of the ratios could not be accurately proved or disproved, inasmuch as none of the plants had complete data to show that the ratios had always been maintained.

Results of tests on embrittlement cracking in Great Britain frequently vary with those obtained by the Bureau. A discussion[52/] of a report on such an investigation showed that the author's findings were not inconsistent with the previously published American work. The same effects of concentrated stress and highly concentrated solutions of pure sodium hydroxide were obtained. The author's results on the inhibiting effects of tannin were also in agreement, insofar, as they went, with our tests.

Laboratory study disclosed that a projected use of a proprietary compound to acid-clean a boiler at a naval installation in Maryland would result in serious deterioration of the boiler; stopping the proposed acid-cleaning operation undoubtedly saved a boiler valued at many thousands of dollars.

Smoke Abatement

Smoke-abatement work throughout the United States continued to be very active. Cities requesting Bureau of Mines publications and consulting service included the following: Victoria, British Columbia; Windsor, Conn.; Elgin, Ill.; Louisville, Ky.; Rumford, Maine; Detroit, Mich.; Weehawken, N. J.; Amsterdam, N. Y.; Dayton, Ohio; Erie, Pa.; Austin, Tex.; and Tacoma, Wash. Consulting and field services were given various Federal agencies on problems of abating smoke and dust, including acceptance tests of new dust-precipitating equipment for the West Central Heating Station in Washington, D. C. The National Smoke Abatement Society of England exhibited American publications and charts on smoke abatement at Manchester, England. Work on smoke-abatement problems for the International Joint Commission was continued by its Technical Advisory Board on Air Pollution, which includes a Bureau of Mines engineer. Studies were made regarding the education of personnel and changes in fuel-burning equipment on vessels of the Great Lakes using the Detroit River.

A historical paper on air-pollution prevention in the United States[53/] gave a history of adoption of ordinances, the emissions prohibited and standards of measurements, the developments in continuing the work, related activities, a discussion of the general nature of the problem, and a list of references. Another paper[54/] presented quantitative data from a number of metallurgical operations on air pollution by industrial fumes, gases, and dusts and discussed allowable stack discharge

52/ Berk, A. A., Discussion of "Caustic Cracking: Stress Corrosion Tests as Elevated Temperatures," by C. C. Weir: Inst. Mech. Eng. (London), War Emerg. Proc., No. 55, 1949, p. 24.

53/ Barkley, J. F., Air-Pollution Prevention in the United States: Mech. Eng., April 1951, vol. 73, No. 4, pp. 284-288.

54/ McCabe, Louis C., Air Pollution by Industrial Fumes, Gases, and Dusts: Am. Inst. Min. and Met. Eng. Tech. Pub. 2915F, Trans. Am. Inst. Min. and Met. Eng., vol. 187, 1950, pp. 971-973; Min. Eng. vol. 187, No. 9, September 1950, pp. 971-973.

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in terms of effluent-weight ratio. Types of equipment used for recovery of stack effluents were cited. A Bureau of Mines information circular covering general aspects of smoke abatement[55] was published. Several model ordinances were included and discussed, with a discussion on smoke abatement at plants.

Coal and Energy in the Western States

A review was made of pertinent technical and economic facts relating to the western coal industry.[56] The demand for energy on the west coast was analyzed, and it was estimated that if the oil produced and imported does not exceed 1,000,000 barrels per day and the natural gas produced and imported does not exceed 2 billion cubic feet per day, the demand for coal will increase to 6 to 8 million tons per year. Since 1920, coal has lost 30 percent of its markets in the Western States due to competition with oil and gas. In 1920 coal supplied 7.1 percent of the energy produced, but at the present time it supplies only about 4 percent. The paper lists the various coal-mining fields and gives the history of coal production of the Western States and averages analyses of the coal from each field. The classification of coal is discussed, and charts are given to correlate the analysis of coal with heating value and classification.

Combustion Characteristics of Packaged Fuels

An investigation was conducted as an extension of previous Bureau studies of packaged fuels containing anthracite fines to determine the combustion characteristics of packaged fuels made of high-volatile and low-volatile bituminous coals.[57] Particular attention was given to the smoke-producing properties of the packaged fuels, because of the demands for the reduction of smoke from hand-fired domestic-heating furnaces.

It was found that packaged fuel of sufficient cold strength for handling can be made if sufficient binder is used. The combustion tests indicated that most of the packaged fuels made from the more strongly caking coals and 4 percent asphalt binder had satisfactory burning properties. The quantity of smoke produced increased with the volatile content of the fuel. Only two of the six different lots of packaged fuels tested would satisfy the smoke ordinances if burned without secondary air, but all except one lot were acceptable if burned with sufficient secondary air.

Factors Affecting Heat Absorption in Furances

To provide a basis for rational and economical design of furnaces and to permit the most economic use of available coals, the Special Research Committee on Furnace Performance Factors of the American Society of Mechanical Engineers continued its cooperation with the Bureau of Mines in investigations to determine the effect of method of firing, load, excess air, and cleanliness of furnace walls on heat absorp- in boiler furnaces.

55/ Barkley, J. F., Fundamentals of Smoke Abatement: Bureau of Mines Inf. Circ. 7588, 1950, 34 pp.

56/ Parry, V. F., Production, Classification, and Utilization of Western United States Coals: Econ. Geol., vol. 45, No. 6, September-October 1950, pp. 515-532.

57/ Myers, James W., and Corey, Richard C., Combustion Characteristics and Physical Properties of Packaged Fuels Containing Bituminous Coal: Bureau of Mines Rept. of Investigations 4797, 1951, 30 pp.

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Heat-absorption studies were completed on two large electric utility boiler furnaces, both pulverized-coal-fired but differing in design and type of firing.[58/59] In both furnaces the heat transfer from the flame was found to be considerably less than that which is theoretically achievable from a perfect radiation source at the temperature of the flame. However, good correlation was noted between the fourth power of the temperature of the gases leaving the furnaces and the heat absorption in the respective furnaces.

Disposal of Radioactive Wastes by Incineration

With increasing use of radioisotopes for research and medical therapy in clinics, hospitals, universities, and other institutions, the care in the disposal of radioactive wastes to prevent contamination of personnel, ground waters, and the atmosphere must be simple and economical. Combustible wastes, such as paper, cloth, wood, animals, etc., comprising the largest part of such wastes, are most conveniently handled by incineration, since a 10- to 15-fold reduction in volume of material is attained. However, ash and other solid incinerator residues and the products of combustion present a special handling problem in densely populated areas, and the Atomic Energy Commission contracted with the Bureau of Mines to develop an incinerator to meet the needs in this respect.

The first phase of this project was completed and consisted of devising a method for collecting dry residues from an incinerator in a manner that would obviate the need for manual handling of the residues, in which the level of radioactivity might be relatively high as compared to the original waste. The method[60/] consists of catching and dissolving the residues in molten sodium hydroxide held at a temperature of 1,000° F. during operation of the incinerator. When the flux is saturated with residues, it can be shipped as a solid, compact mass to the final disposal point. It is estimated that a 100-pound charge of sodium hydroxide will handle the residue from approximately 2,000 pounds of waste material. Owing to the low cost of the flux and the use of disposable plain carbon steel pots to retain the flux, the cost of this operation would be a negligible factor in the over-all operating costs. Work is being continued on the design and construction of the incinerator and auxiliary equipment.

CARBONIZATION OF COAL

Survey of Carbonizing Properties of American Coals

Pilot-scale carbonization tests at high temperatures only were continued as during the past 2 years. As this work comprises the third (carbonization) phase of the coking-coal reserve investigation, emphasis was placed on the suitability of the coals tested for making metallurgical coke; however, yields and quality of other carbonization products were also determined. Most coals were carbonized singly and in blends in order to evaluate more completely their coke-making properties.[61/]

58/ Corey, R. C., and Cohen, P., Furnace Heat Absorption in Paddy's Run Pulverized-Coal-Fired Steam Generator, Using Turbulent Burners, Louisville, Ky.: Trans. Am. Soc. Mech. Eng., vol. 72, No. 7, October 1950, pp. 925-935.

59/ Myers, J. W., and Corey, R. C., Furnace Heat Absorption in Pulverized-Coal-Fired Steam Generator, Willow Island Station: Trans. Am. Soc. Mech. Eng., vol. 73, May 1951, pp. 419-431.

60/ Corey, R. C., Perry, H., and Schwartz, C. H., Off-Site Disposal of Radioactive Incinerator Residues by Solid Fluxes: Am. Ind. Hygiene Assoc. Quarterly, vol. 12, No. 2, June 1951, pp. 52-57.

61/ Davis, J. D., Survey of Carbonizing Properties of American Coals, Bureau of Mines Coal-Carbonization Laboratory, 1949-50: Proc. Am. Gas Assoc., 1950, pp. 402-409.

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TABLE 8. - Description and analysis of coals and proportions used in blends

Coal No.	Description	Analysis, percent					
		Proximate					Dry, mineral matter-free fixed carbon
		H_2O	Volatile matter	Fixed carbon	Ash	Sulfur	
West Virginia							
449.....	Beckley bed, Caretta No. 5 mine, Susanna, McDowell County.....	3.1	15.8	72.4	8.7	0.7	83.0
449A.....	Blend: 20 percent Beckley (449) and 80 percent Pittsburgh (r28).	-	-	-	-	-	-
449B.....	Blend: 30 percent Beckley (449) and 70 percent Pittsburgh (r28).	-	-	-	-	-	-
450.....	Beckley bed, Glen Rogers No. 2 mine, Glen Rogers, Wyoming County.	1.6	18.5	75.4	4.5	1.1	80.8
450A.....	Blend: 20 percent Beckley (450) and 80 percent Pittsburgh (r28).	-	-	-	-	-	-
450B.....	Blend: 30 percent Beckley (450) and 70 percent Pittsburgh (r28).	-	-	-	-	-	-
465.....	Sewell bed, Imperial No. 3 mine, Quinwood, Greenbrier County.....	2.8	27.1	66.8	3.3	.5	71.4
465A.....	Blend: 20 percent Sewell (465) and 80 percent Pittsburgh (r28)..	-	-	-	-	-	-
465B.....	Blend: 30 percent Sewell (465) and 70 percent Pittsburgh (s28)..	-	-	-	-	-	-
465C.....	Blend: 50 percent Sewell (465) and 50 percent Pittsburgh (s28)..	-	-	-	-	-	-
466.....	Pittsburgh bed, Morgan No. 2 mine, Perry County.....	3.0	36.4	54.4	6.2	1.3	60.4
467.....	Winifrede bed, Handley No. 7 mine, Kanawha County.....	2.9	34.1	58.8	4.2	.5	63.6
467A.....	Blend: 80 percent Winifrede (467) and 20 percent Pocahontas (h75).....	-	-	-	-	-	-
467B.....	Blend: 70 percent Winifrede (467) and 30 percent Pocahontas (h75).....	-	-	-	-	-	-
467C.....	Blend: 90 percent Winifrede (467) and 10 percent Pocahontas (h75).....	-	-	-	-	-	-
467D.....	Blend: 75 percent Winifrede (467) and 25 percent Pocahontas (h75).....	-	-	-	-	-	-
468.....	Winifrede bed, Crown Hill No. 4 mine, Kanawha County.....	2.4	35.6	57.8	4.2	.7	62.2
470.....	Upper Freeport bed, Sago mine, Sago, Upshur County.....	3.3	38.1	53.5	5.1	2.0	58.7
479.....	Pocahontas No. 3 bed.....	6.0	17.3	71.3	5.4	.8	81.0
Pennsylvania							
442.....	Lower Freeport bed, Hess and Hess mine, Dixonville, Indiana County.....	2.5	29.3	57.7	10.5	2.9	67.4
442A.....	Blend: 70 percent Lower Freeport (442) and 30 percent Lower Kittanning (443).....	-	-	-	-	-	-
442B.....	Blend: 80 percent Lower Freeport (442) and 20 percent Lower Kittanning (443).....	-	-	-	-	-	-
442C.....	Blend: 80 percent Lower Freeport (442) and 20 percent Pocahontas (h75).....	-	-	-	-	-	-
443.....	Lower Kittanning bed, McKain mine, Starford, Indiana County.....	3.3	27.3	61.7	7.7	2.1	70.1
444.....	Thick Freeport bed, Russelton No. 2 mine, Allegheny County.....	6.0	32.5	52.2	9.3	.9	62.4
445.....	Thick Freeport bed, Russelton No. 2 mine, Allegheny County.....	4.4	16.1	16.8	62.7	8.3	65.1
448.....	Upper Freeport bed, Decker No. 2 mine, Armstrong County.....	3.2	34.1	56.0	6.7	1.2	62.7
451.....	Lower Kittanning bed, Armstrong County.....	1.5	38.6	52.1	7.8	2.5	58.1
452.....	Lower Kittanning bed, Cadogan mine, Armstrong County.....	3.5	24.2	28.4	43.9	21.0	61.6
458.....	Upper Freeport bed, Rose's mine, Dunbar, Fayette County.....	2.6	31.2	57.6	8.6	2.1	65.7
458A.....	Blend: 80 percent Upper Freeport (458) and 20 percent Pocahontas (h75).....	-	-	-	-	-	-
458B.....	Blend: 70 percent Upper Freeport (458) and 30 percent Pocahontas (h75).....	-	-	-	-	-	-
469.....	Sewickley bed, Atlas mine, Fayette County.....	2.9	32.2	53.4	11.5	3.2	63.5
469A.....	Blend: 80 percent Sewickley (469) and 20 percent Pocahontas (h75).....	-	-	-	-	-	-
469B.....	Blend: 70 percent Sewickley (496) and 30 percent Pocahontas (h75).....	-	-	-	-	-	-
480.....	Upper Freeport bed, Delmont mines 10 and 10a, Hunkers, Westmoreland County.....	1.7	32.8	56.2	9.3	3.3	64.1
480A.....	Blend: 80 percent Upper Freeport (480) and 20 percent Pocahontas (h75).....	-	-	-	-	-	-
480B.....	Blend: 70 percent Upper Freeport (480) and 30 percent Pocahontas (h75).....	-	-	-	-	-	-

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TABLE 8. - Description and analysis of coals and proportions used in blends (Cont.)

Coal No.	Description	Analysis, percent					
		Proximate					Dry, mineral matter-free fixed carbon
		H_2O	Volatile matter	Fixed carbon	Ash	Sulfur	
	Virginia						
476.....	Tiller bed, Shickler mine, Dickenson County.....	2.5	26.3	67.9	3.3	0.6	72.4
478.....	Clintwood bed, from near Gladeville, Wise County.....	3.3	31.1	58.9	6.7	.6	66.0
	Tennessee						
456.....	Jellico bed, Blue Rose mine, Morely, Campbell County.....	2.7	37.3	55.8	4.2	1.0	60.3
456A.....	Blend: 80 percent Jellico (456) and 20 percent Pocahontas (g75).	-	-	-	-	--	-
456B.....	Blend: 70 percent Jellico (456) and 30 percent Pocahontas (g75).	-	-	-	-	--	-
477.....	Sewanee bed, Reels Cove No. 2 mine, Whitwell, Marion County.....	2.3	28.5	60.6	8.6	.7	68.7
477A.....	Blend: 60 percent Sewanee (477) and 40 percent Pocahontas (479).	-	-	-	-	-	-
477B.....	Blend: 70 percent Sewanee (477) and 30 percent Clintwood (478)..	-	-	-	-	-	-
477C.....	Blend: 70 percent Sewanee (477) and 30 percent Pittsburgh (s28).	-	-	-	-	-	-
477D.....	Blend: 50 percent Sewanee (477) and 50 percent Pittsburgh (s28).	-	-	-	-	-	-
477E.....	Blend: 30 percent Sewanee (477) and 70 percent Pittsburgh (s28).	-	-	-	-	--	-
	Alabama						
475.....	Blend: 50 percent Black Creek and 50 percent Mary Lee beds.....	-	-	-	-	-	-
	Washington						
446.....	Wilkeson No. 3 bed.....	3.8	32.1	53.4	10.7	0.4	63.3
447.....	Wilkeson No. 2 bed.....	2.0	31.0	55.3	11.7	.4	65.0
	South America						
434.....	Veta Principal bed, Cundiamarca Dept., Colombia.....	1.9	33.5	50.0	14.6	3.5	61.2
434A.....	Blend: 50 percent coal 434 and 50 percent coal 435.....	-	-	-	-	-	-
435.....	Veta Secundaria bed, Cundiamarca Dept., Colombia.....	2.0	20.0	63.4	14.6	1.1	75.5
436.....	No. 1 bed, Valle del Cauca Dept., Colombia.....	4.7	27.2	55.5	12.6	2.6	68.4
437.....	Grande bed, Valle del Cauca Dept., Colombia.....	4.9	22.0	67.4	5.7	.8	75.9
438.....	Nos. 5 and 6 beds, Valle del Cauca Dept., Colombia.....	4.6	40.1	42.6	12.7	3.4	52.5
439.....	Grande Nos. 2 and 3 beds, Valle del Cauca Dept., Colombia.....	4.4	37.5	50.6	7.5	2.7	58.1
440.....	Nos. 3 and 4 beds, Valle del Cauca Dept., Colombia.....	1.5	41.9	40.8	15.8	.7	50.3
440A.....	Blend: 50 percent coal 440 and 50 percent coal 441						
441.....	Nos. 3 and 4 beds, Valle del Cauca Dept., Colombia.....	2.8	36.7	33.9	26.6	.9	50.0
459.....	Large bed, Valle del Cauca Dept., Colombia.....	2.3	42.1	40.7	14.9	1.1	50.1
460.....	Grande bed, Valle del Cauca Dept., Colombia.....	1.5	41.5	45.5	7.9	.7	50.7
461.....	Mixture of 4 beds, Valle del Cauca Dept., Colombia.....	3.4	39.8	40.3	16.5	.5	51.4
462.....	bed, Valle del Cauca Dept., Colombia.....	5.3	39.5	41.7	13.5	2.5	52.4
463.....	Nos. 2 and 3 beds, Valle del Cauca Dept., Colombia.....	4.0	39.6	41.0	15.4	2.5	52.0
464.....	La Ducha bed, Valle del Cauca Dept., Colombia.....	5.1	21.5	64.5	8.9	1.7	75.9
	Miscellaneous						
471.....	Lignite, North Dakota.....	37.0	25.6	28.5	8.9	0.4	53.6
473.....	Sandow lignite, Milam County, Tex.	28.4	31.8	29.8	10.0	1.3	49.3
481.....	Pittsburgh No. 8 bed, Jefferson County, Ohio.....	5.0	38.0	49.5	7.5	2.7	57.2
482.....	Pittsburgh No. 8 bed, Belmont County, Ohio.....	5.2	39.5	47.7	7.6	3.7	55.4
	Blending coals						
r28.....	Pittsburgh bed, Warden mine, Allegheny County, Pa.	3.1	33.0	54.6	9.3	1.2	63.1
s28.....	Pittsburgh bed, Warden mine, Allegheny County, Pa.	1.8	35.4	57.7	5.1	.9	62.4
h75.....	Pocahontas No. 3 bed, McDowell County, W. Va.	2.7	16.8	74.1	6.4	.6	82.2

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TABLE 9. - Physical properties of coke

A.S.T.M. METHOD

Coal No.	Carbonizing temperature, °C.	True specific gravity	Apparent specific gravity	Cells, percent	Shatter test,1/ cumulative percent upon- 2-inch screen	1-1/2-inch screen	1-inch screen	1/2-inch screen	Tumbler test,1/ cumulative percent upon- 2-inch screen	1-1/2-inch screen	1-inch screen	1/2-inch screen	1/4-inch screen
West Virginia													
449A.....	800	1.88	0.84	55.3	90	95	97	98	18	46	56	59	59
449B.....	800	1.86	.84	54.8	91	96	97	98	21	50	62	64	64
449.....	900	1.90	.92	51.6	64	91	98	99	3	24	56	63	64
449A.....	900	1.92	.85	55.7	69	90	96	97	1	19	51	61	62
449B.....	900	1.89	.86	54.5	68	90	96	98	2	23	50	60	62
450A.....	800	1.86	.87	53.2	88	95	97	98	16	45	59	62	63
450B.....	800	1.85	.85	54.1	91	96	97	98	22	53	62	64	64
450.....	900	1.87	.87	53.5	72	93	97	99	4	30	58	64	65
450A.....	900	1.89	.87	54.0	63	86	96	98	2	22	53	64	66
450B.....	900	1.89	.86	54.5	71	90	97	98	3	24	56	66	67
465B.....	800	1.83	.85	53.6	81	92	96	98	6	32	52	59	60
465C.....	800	1.84	.86	53.3	80	91	96	98	8	30	50	58	59
465.....	900	1.85	.80	56.8	56	86	96	98	2	21	54	63	64
465B.....	900	1.87	.89	52.4	50	83	95	97	0	11	46	61	63
465C.....	900	1.84	.88	52.2	42	84	95	98	0	11	47	63	65
467.....	800	1.83	.85	53.6	75	88	93	96	2	19	38	51	56
467A.....	800	1.85	.85	54.1	84	94	97	98	14	44	57	60	61
467B.....	800	1.85	.84	54.6	86	95	97	98	14	49	58	61	61
467.....	900	1.87	.87	53.5	46	74	93	97	0	11	42	62	65
467A.....	900	1.88	.84	55.3	47	84	96	98	0	14	49	65	67
467B.....	900	1.88	.87	53.7	54	82	93	96	0	14	50	66	67
468.....	900	1.87	.86	54.0	48	77	92	97	0	7	35	57	63
470.....	900	1.85	.82	55.7	48	80	92	96	0	1	22	41	47
Pennsylvania													
442.....	900	1.93	0.91	52.8	68	89	95	97	0	15	41	54	57
442A.....	900	1.93	.87	54.9	68	89	95	97	1	24	47	57	59
442B.....	900	1.93	.87	54.9	68	89	96	97	2	22	48	56	58
442C.....	900	1.92	.88	54.2	68	91	97	98	3	26	51	58	60
458.....	800	1.89	.86	54.5	79	92	96	98	5	29	46	56	59
458A.....	800	1.87	.84	55.1	85	95	98	98	12	43	56	58	59
458B.....	800	1.87	.85	54.5	89	95	97	98	21	48	57	58	58
458.....	900	1.91	.88	53.9	57	86	95	97	1	18	47	58	60
458A.....	900	1.90	.85	55.3	63	90	96	98	3	26	54	61	62
458B.....	900	1.90	.85	55.3	68	90	97	98	3	29	54	61	61
469.....	800	1.92	.95	50.5	82	91	95	97	4	21	38	49	51
469A.....	800	1.91	.96	49.7	87	96	97	98	11	30	53	57	58
469.....	900	1.94	.85	56.2	62	87	95	97	0	9	35	49	52
469A.....	900	1.94	.92	52.6	64	88	96	98	1	11	40	52	54
469B.....	900	1.95	.95	51.3	64	87	96	98	0	16	46	57	59
480.....	800	1.91	.98	48.7	78	90	94	96	3	20	36	50	54
480A.....	800	1.92	.97	49.5	81	93	96	97	7	33	50	57	58
480B.....	800	1.90	.93	51.1	90	95	97	98	16	44	56	59	59
480.....	900	1.93	.91	52.8	58	87	95	97	0	10	40	55	57
480A.....	900	1.94	.96	50.5	60	87	96	97	1	15	46	57	59
480B.....	900	1.93	.91	52.8	64	87	96	98	1	20	49	59	61

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TABLE 9. - Physical properties of coke (Cont.)

Coal No.	Carbonizing temperature, °C.	True specific gravity	Apparent specific gravity	Cells, percent	Shatter test,1/ cumulative percent upon- 2-inch screen	Shatter test,1/ cumulative percent upon- 1-1/2-inch screen	Shatter test,1/ cumulative percent upon- 1-inch screen	Shatter test,1/ cumulative percent upon- 1/2-inch screen	Tumbler test,1/ cumulative percent upon- 2-inch screen	Tumbler test,1/ cumulative percent upon- 1-1/2-inch screen	Tumbler test,1/ cumulative percent upon- 1-inch screen	Tumbler test,1/ cumulative percent upon- 1/2-inch screen	Tumbler test,1/ cumulative percent upon- 1/4-inch screen
Tennessee													
456.....	800	1.82	0.81	55.5	62	77	89	96	0	3	21	47	59
456A.....	800	1.84	.83	54.9	84	93	96	97	8	35	53	59	60
456B.....	800	1.82	.82	54.9	88	95	97	98	12	42	58	61	61
456.....	900	1.86	.81	56.5	49	79	91	96	0	4	28	52	60
456A.....	900	1.86	.86	53.8	47	82	95	98	0	10	47	65	67
456B.....	900	1.86	.84	54.8	51	82	95	97	1	10	48	65	67
477.....	800	1.88	.84	55.3	83	94	97	98	9	47	61	64	64
477D.....	800	1.87	.85	54.5	78	91	96	97	6	29	48	63	64
477E.....	800	1.86	.86	53.8	74	87	95	97	3	21	43	62	64
477.....	900	1.90	.84	55.8	57	85	96	98	1	22	57	66	67
477A.....	900	1.90	.83	56.3	61	89	97	98	4	25	57	66	66
477B.....	900	1.90	.84	55.8	59	87	96	97	2	23	55	65	67
477C.....	900	1.90	.86	54.7	52	85	96	98	1	22	58	68	69
477D.....	900	1.90	.86	54.7	47	85	95	97	1	18	54	67	68
477E.....	900	1.89	.88	53.4	38	79	95	98	0	10	52	67	68
477.....	1,000	1.92	.85	55.7	26	69	94	98	2	6	46	69	70
Alabama													
475.....	800	1.88	0.92	51.3	88	94	97	98	11	44	57	61	62
475.....	900	1.91	.90	52.8	55	86	96	98	0	15	50	62	64
475.....	1,000	1.93	.90	53.4	24	63	93	98	1	3	36	63	65

BM-AGA METHOD

Washington													
446.....	900	1.94	0.79	59.3	41	79	95	98	0	19	53	-	63
447.....	900	1.93	.87	54.9	25	76	94	98	0	18	64	-	75
South America													
434.....	900	1.98	0.90	54.5	47	78	93	97	0	20	56	-	67
434A.....	900	1.94	.95	51.0	67	89	96	98	8	39	64	-	71
435.....	900	1.96	.93	52.6	62	88	96	98	11	42	65	-	72
436.....	900	1.99	.78	60.8	38	84	95	98	3	30	60	-	68
437.....	900	1.90	.81	57.4	49	83	95	99	4	30	65	-	78
438.....	900	2.02	.83	58.9	37	66	85	96	0	5	35	-	65
439.....	900	1.94	.74	61.9	28	64	91	97	0	9	46	-	67
440A.....	900	2.06	.89	56.8	68	84	92	96	3	19	35	-	43
459.....	900	2.00	.81	59.5	30	55	76	94	0	1	18	-	60
460.....	900	1.93	.76	60.6	6	41	69	97	0	0	10	-	71
461.....	900	2.01	.80	60.2	42	70	86	95	0	6	34	-	55
462.....	900	1.99	.81	59.3	31	65	85	97	0	2	31	-	63
463.....	900	2.04	.80	60.8	42	78	90	97	1	13	46	-	67
464.....	No tests made; coke was mostly char.												

1/ The actual sizes of screens designated at 1, 1/2, and 1/4 inch were 1.06, 0.53, and 0.26 inch, respectively.

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Samples from 15 coal beds of the Appalachian region, 2 from Washington, and 14 from Colombia, South America, were tested. The source, proximate analysis, and sulfur content of the coals used and the composition of the blends are given in table 8, which also includes additional samples obtained for plasticity, oxidation, or expansion tests. The Appalachian region coals were tested at 800° and 900° C. in the standard 18-inch retort by the Bureau of Mines-American Gas Association (BM-AGA) methods. Physical properties of the cokes from these tests were determined by standard methods of the American Society for Testing Materials. The Washington and South American coals were tested at 900° C. in the BM-AGA standard 13-inch retort and the cokes by BM-AGA methods, wherein the tumbler test is less severe than the A.S.T.M. method. Expanding properties were measured largely by tests in the Bureau of Mines sole-heated oven. Plastic properties were determined by the Gieseler and Davis methods. Physical properties of the coke, which are primarily important in judging the suitability of a coal or blend for commercial carbonization, are given in table 9, by States, and are summarized below.

West Virginia Coals

Two low-volatile Beckley-bed coals (449 and 450) blended with 80 percent high-volatile Pittsburgh-bed coal gave indexes for the 900° C. cokes that show both coals are suitable for blending with high-volatile coking coals for producing metallurgical coke. Raising the proportion of these Beckley coals to 30 percent did not benefit the coke from the Caretta blend, although some improvement was noted for the Glen Rogers blend. If blended correctly, they could be substituted for Pocahontas No. 3 in coking blends without loss in coke quality.

Sewell-bed coal (465), medium-volatile bituminous in rank, coked strongly when carbonized alone; however, it should be blended with lower-ranking coal for commercial carbonization, because it expands during carbonization. Its blends (465C and 465B) with 50 and 70 percent Pittsburgh-bed coal yielded strong coke at 900° C. The 50:50 blend of this Sewell and of Pittsburgh coal yielded coke of metallurgical grade, and even greater proportions of Sewell coal probably could be used satisfactorily.

Winifrede-bed coals (467 and 468), high-volatile A bituminous in rank, yielded well-fused, moderately fissured coke at 900° C.; the coke from No. 7-mine coal was less abradable. The 80:20 blend of No. 7-mine and Pocahontas No. 3 coals gave strong coke at 900° C. Increasing the proportion of Pocahontas No. 3 to 30 percent did not significantly benefit the coke. Either blend should be suitable for the production of metallurgical coke.

Upper Freeport-bed coal (470), high-volatile A bituminous in rank, yielded rather spongy coke, and, as would be expected, the 1- and 1/4-inch tumbler indexes were low. This coal attains a high fluidity in the plastic state; probably it could be used to produce metallurgical coke if blended with low-sulfur coals of higher rank and lower fluidity.

In addition to the coals listed in table 8, carbonization tests were reported on the Eagle, No. 2 Gas, Pocahontas No. 3, and Pocahontas No. 4 beds from West Virginia.62/ These four coals, the first two of high-volatile A rank and the last two low-volatile, respectively, were found to be suitable for the manufacture of oven coke if blended in proper proportions. The blends should be carbonized at moderate bulk densities, since they may expand enough at high densities to damage oven walls.

62/ Davis, J. D., Reynolds, D. A., Brewer, R. E., Ode, W. H., Naugle, B. W., Wolfson, D. E., Birge, G. W., Carbonizing Properties: West Virginia Coals from the Eagle, No. 2 Gas, Pocahontas No. 3, and Pocahontas No. 4 Beds: Bureau of Mines Bull. 493, 1950, 39 pp.

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The carbonizing properties of an additional medium-volatile and five low-volatile coals from West Virginia were reported, with data on three Pennsylvania coals.63/ The low-volatile coals comprised two samples of Pocahontas No. 6-bed coal from Wyoming County and one from Mercer County, Davy Sewell bed-coal from McDowell County, and Fire Creek bed-coal from Greenbrier County. The medium-volatile coal was from the Fire Creek bed, Greenbrier County. Pittsburgh-bed coal from the Warden mine, Allegheny County, Pa., was used in blends with these coals. All samples coked strongly when carbonized singly. The blends containing 80 percent Pittsburgh also coked strongly. The cokes made from these blends compared favorably with those made from the BM-AGA standard blend of coals from Pittsburgh and Pocahontas No. 3 beds. Davy Sewell, Pocahontas No. 6, and Fire Creek low-volatile coals were nearly as suitable for blending with Pittsburgh coal as Pocahontas No. 3. The medium-volatile Fire Creek coal was slightly less suitable, but its blends would probably coke more strongly if it were used in larger proportions.

Pennsylvania Coals

Lower Freeport-bed coal (442), of high-volatile A rank, yielded strong coke when blended with 20 percent medium-volatile Lower Kittanning (443) or 20 percent Pocahontas No. 3 (h75) coals; however, the hardness factors, or 1/4-inch tumbler indexes, of the blend cokes were slightly low at 58 and 60. The coking property of the Lower Freeport-Lower Kittanning blend was not improved by raising the proportion of Lower Kittanning from 20 to 30 percent.

Lower Kittanning-bed coal (443) ranked low in the medium-volatile group. It was carbonized only as blends (442A and 442B) with Lower Freeport coal. Lower Kittanning is a coking coal which should be so blended that the coal rank is changed little.

Upper Freeport-bed coal (458), high-volatile A bituminous in rank, was cleaned by the heavy media process to yield a product containing 8.7 percent ash and 2.1 percent sulfur. The 900° C. coke was well-fused and strong for coal of this rank. Blending with 20 percent Pocahontas No. 3 raised the 1-inch tumbler index from 47 to 54; other indexes were raised less. The tests indicate that 80 percent is about the optimum proportion of this coal to be blended with Pocahontas No. 3.

Another high-volatile A Upper Freeport-bed coal (480) gave 900° C. coke that resisted breakage well in the shatter test but was rather abradable in the tumbler test. Blending with 20 percent Pocahontas No. 3 benefited the coke by raising its 1- and 1/4-inch tumbler indexes from 40 to 46 and from 57 to 59, respectively. The 70:30 blend of these coals yielded slightly stronger coke. Although washed, the high sulfur content of this coal would make it unsuited for metallurgical use.

The three Pennsylvania coals, included with the report on West Virginia coals64/ previously mentioned, were medium-volatile coals from the Lower Kittanning bed, Cambria County, and the Upper Kittanning bed, Clearfield County, and high-volatile A coal from the Upper and Lower Freeport beds, Indiana County. All of these coals coked strongly when carbonized alone. The medium-volatile coals, when blended with standard Pittsburgh-bed blending coal, did not make quite as strong coke as blends of low-volatile coal with the Pittsburgh coal, but probably such blends would be stronger if larger proportions of the medium-volatile coals were used.

63/ Davis, J. D., Reynolds, D. A., Brewer, R. E., Wolfson, D. E., Naugle, B. W., and Birge, G. W., Carbonizing Properties: Pocahontas No. 6, Davy Sewell, and Fire Creek Coals from West Virginia and Upper and Lower Kittanning and Upper and Lower Freeport Coals from Pennsylvania: Bureau of Mines Bull. 496, 1950, 42 pp.

64/ See footnote 63.

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Virginia Coals

Clintwood-bed coal (478) from an unknown mine near Gladeville, Wise County, Va., ranked high in the high-volatile A classification. As a blend (477B) with 70 percent Sewanee, it coked strongly. The coal was not tested singly.

Tennessee Coals

Jellico-bed coal (456), high-volatile A bituminous in rank, gave rather weak coke, but was improved markedly by blending with 20 and 30 percent Pocahontas No. 3. Either of these blends should yield metallurgical coke in commercial ovens, although the blend containing 30 percent Pocahontas cokes more strongly.

Sewanee-bed coal (477), ranking at the top of the high-volatile A classification, coked strongly when carbonized singly. The 60:40 Sewanee-Pocahontas No. 3 blend (477A), which is carbonized commercially to produce foundry coke, yielded coke that resisted breakage in the shatter test more than the Sewanee coke. The 70:30 Sewanee-Clintwood blend (477B) coked nearly as strongly as Sewanee. Substitution of an equal amount of Pittsburgh-bed coal for Clintwood lowered the shatter indexes and raised the tumbler indexes, although the changes were small.

Washington Coals

Wilkeson No. 2- and No. 3-bed coals (447 and 446), washed at the Bureau station at Seattle, gave cokes that were stronger than the average coke from coals of similar (high-volatile A) rank, except that the No. 3 coke had a hardness factor of 63, whereas the average is 72.

Alabama Coals

A 50:50 blend (475) of Black Creek- and Mary Lee-bed (medium-volatile washed coals from Jefferson County), carbonized at 800°, 900°, and 1,000° C., yielded cokes that were strong at 800° and 900° C. The 1,000° C. coke was unstable in the shatter and tumbler tests because it was more fissured.

Colombia, South America, Coals

The Colombian coals ranged from low in the high-volatile A rank to high in the medium-volatile. High-volatile A Principal-bed coal (434) yielded strong coke that compared favorably with coke made from eastern domestic coals of similar rank. Medium-volatile Secundaria-bed coal (435) yielded the strongest coke; this coke was almost as strong as cokes made from the best coking coals of West Virginia. A 50:50 blend (434A) of these two coals, both from the Department of Cundiamarca, coked almost as strongly as 100 percent Secundaria coal. These coals are suitable for the production of metallurgical coke if blended properly to eliminate the possibility of expansion during carbonization.

Only two coals from the department of Valle del Cauca yielded strong coke. The No. 1 (436) and Grande (437) beds, ranked as high-volatile A bituminous, gave cokes that were satsifactorily strong, considering their rank. Blending with coals of low rank would benefit the coking of these coals.

The following coals from the Department of Valle del Cauca yielded rather weak cokes: No. 5 and No. 6 bed-blend (438), Grande beds Nos. 2 and 3 (439 and 463), No. 3 and No. 4 beds (440), Large bed (459), Grande bed (460), four mixed beds,

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(461), and an unknown bed (462). Most of these cokes were hard and probably could be strengthened by blending the coals to increase their rank. The coking properties of six samples, containing 13.5 to 26.6 percent ash, probably would be benefited by cleaning the coals to moderate ash content. La Ducha-bed coal (464) did not yield coherent coke, although it ranked high in the medium-volatile group.

Comparison of BM-AGA and Slot-oven Experimental Methods of Carbonization

A comparison was made between carbonizing tests in the BM-AGA cylindrical steel retorts and in a vertical, refractory slot-oven similar to that developed by the Illinois State Geological Survey.65/ Three high-volatile coals and eight blends of coals, differing in rank, were carbonized in BM-AGA retorts at 800° and 900° C. and in the slot oven at 870° to 1,010° C. Average yields from BM-AGA tests at 800° and 900° C. and the slot oven, respectively, were: Coke, percent, 70.5 70.0, and 70.5; gas, cubic feet per ton, 8,900 10,100, and 9,650; tar, gallons per ton, 13.0, 11.4, and 9.0; light oil, gallons per ton, 1.95, 2.39, and 1.75; and ammonium sulfate, pounds per ton, 27.2, 23.6, and 22.3, The slot-oven and 900° C. cokes were of similar chemical composition; the 800° C. cokes contained slightly more volatile matter and less fixed carbon. Generally, other physical properties of the slot-oven cokes were intermediate between those of the 800° and 900° C. BM-AGA cokes. The specific gravity of the gas from the slot-oven generally was intermediate between those of the gases from the 800° and 900° C. BM-AGA tests; heating values were lowest for the slot-oven gas. The tars from the 900° C. BM-AGA and slot-oven tests were similar; they differed significantly only in their content of solids (naphthalene, anthracene, and pitch), which was higher for the slot-oven tar. The light oils from the slot-oven tests contained greater proportions of benzene and lower proportions of paraffins and solvent naphtha.

Small-Scale Laboratory Carbonization Tests

Small-scale laboratory coking tests, made to obtain information on the chemical and physical properties of coals that affect yields and properties of the gas, coke, and coal chemicals, included low-temperature carbonization (Fischer) assays, agglutinating value tests, and free-swelling tests.

Data from these tests on Pocahontas No. 3-, Pocahontas No. 4-, Eagle-, and the No. 2 Gas-bed coals were published66/ to supplement BM-AGA tests at 800° and 900° C. and other tests designed to evaluate plastic and expanding properties of the coals.

Plasticity of Coals

Plastic properties of 39 coals and 34 blends were determined during the fiscal year. Table 8 describes these coals and blends, except blending coal g75, which is similar to coal h75, and special samples of blends, pretreated coals, and sized fractions. All samples were tested by the Gieseler and/or Davis plastometer methods. A total of 383 tests - 197 by the Gieseler and 185 by the Davis method - was made.

Table 10 lists the numbers of the coals and blends tested, the Gieseler maximum fluidity values in dial divisions per minute, and the Davis maximum resistance values in pound-inches. All coals and blends, except the special samples (not listed), were tested in connection with the BM-AGA survey of their carbonizing and expanding properties. The plastic properties of the coals and blends are summarized under "Remarks" in the table.

65/ Davis, J. D., Reynolds, D. A., Wolfson, D. E., and Birge, G. W., Comparison of BM-AGA and Slot-Oven Experimental Methods of Carbonization, with Results for Eleven Coals: Bureau of Mines Bull. 480, 1950, 37 pp.

66/ See footnote 62.

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TABLE 10. - Plastic properties of coals and blends

Coal No.	Gieseler[1]	Davis[2]	Remarks
West Virginia Coals and Blends			
449	(3)	29	Increased Gieseler fluidity and decreased Davis resistance values (blends 449A, 449B, 450A, and 450B) result by blending low-volatile Pocahontas No. 3 coals (449 and 450) with 80 and 70 percent high-volatile A Pittsburgh coal (r28). Reverse trends (blends 465A, 465B, and 465C) are shown by blending medium-volatile Sewell coal (465) with 80, 70, and 50 percent Pittsburgh coal. Top of bed of Pittsburgh coal (466) was excluded, and data are not representative of bed. Blending high-volatile A Winifrede coal (467) with 20 and 30 percent Low-volatile Pocahontas No. 3 coal (h75) decreases fluidity and increases resistance values. High-volatile A Winifrede coal (468) shows higher fluidity and higher resistance than Winifrede coal (467). High-volatile A Upper Freeport coal (470) gives extremely high fluidity and average resistance. Pocahontas No. 3 coal (479) shows plastic characteristics typical of its low-volatile rank.
449A	600	18.5	
449B	830	29	
450	55	62	
450A	450	35	
450B	315	27	
465	1,950	70	
465A	735	12.8	
465B	1,025	39	
465C	1,260	54	
466	(4)	9.8	
467	1,640	11.8	
467A	465	35	
467B	150	35	
468	5,640	15	
470	60,000	10.5	
479	29	72	
Pennsylvania Coals and Blends			
442	30,000	12.3	Gieseler fluidity values cover a wide range. Davis resistance values are normal and indicate good coke-making properties. Except for medium-volatile Lower Kittanning coal (443) and washed Upper Freeport coal, (448) which cannot be classified, all others are high-volatile A in rank. Upper Freeport coal (448) is much less fluid than the Upper Freeport coal (458). Excepting blends 442A and 442B containing coal 443, all others contain 20 and 30 percent low-volatile Pocahontas No. 3 coal (h75). Gieseler values for blends 442B, 458A, and 469A are unexpectedly high. All other blends show expected changes in both fluidity and resistance on blending.
443	(4)	12	
442A	3,765	9	
442B	60,000	10.5	
442C	(4)	12	
448	13	9	
458	5,725	12.5	
458A	6,330	19	
458B	5,500	11.3	
469	6,330	12.3	
469A	13,500	31	
480	60,000	9	
480A	60,000	11.8	
480B	15,670	37	
Virginia Coals			
476	2,000	-	Medium-volatile Tiller coal (476) and high-volatile A Clintwood coal (478) show values consistent with the rank of the coals and indicate good coke-making properties.
478	8,570	21	
Tennessee Coals and Blends			
456	215	7.5	Decreased fluidity and increased resistance (blends 456A and 456B) show effect of blending 20 and 30 percent low-volatile coal (g75) with high-volatile A coal (456), also shown by 477A with 40 percent low-volatile coal (479) and high-volatile A coal (477). Thirty percent high-volatile A Clintwood coal (478) is more effective than Pittsburgh coal (s28) in increasing fluidity; 50 and 70 percent of latter decrease maximum resistance, indicating weak coke.
456A	125	60	
456B	150	41	
477	600	30	
477A	100	27	
477B	1,290	35	
477C	780	37	
477D	3,430	12.8	
477E	5,450	13	
Alabama Blend			
475	3,160	23	50:50 blend gives expected values.
Washington Coals			
446	(4)	12.5	Data indicate fair coke-making properties.
447	(4)	11.5	Do.
Colombia, South America, Coals and Blends			
434	30,000	12.8	The plastic properties of the 14 coals and 2 blends vary widely. Eight coals (435, 437, 438, 439, 461, 462, 463, and 464) do not fuse at the normal rate of heating of 3° C. per minute in the Davis tests and develop only low fluidity in the Gieseler tests. Two coals (434) and (440) give high fusion; two blends (434A and 440A), containing equal parts of coals 434 and 435 and of 440 and 441, respectively, and four coals (436, 441, 459, and 460) show lower fusion. These six coals and two blends indicate fair coke-making properties which could be improved by judicious blending.
435	1.5	(5)	
434A	3.1	63.5	
436	6.4	13	
437	0.6	(5)	
438	2.4	(5)	
439	1.5	(5)	
440	1,450	8	
441	(6)	12	
440A	295	7.3	
459	280	6	
460	380	7.5	
461	340	(5)	
462	195	(5)	
463	295	(5)	
464	(4)		
Blending Coals			
r28	2,070	-	The high-volatile A blending samples (r28 and s28) show good fusion, typical of this coal rank. The low-volatile blending samples (g75 and h75) show characteristic values. Coals 28 and 75, when used in proper proportions, are excellent blending coals.
s28	2,730	11	
g75	1.9	(5)	
h75	5.3	8.8	

1/ Gieseler plastometer, maximum fluidity in dial divisions per minute.
2/ Davis plastometer, maximum resistance in pound-inches.
3/ No dial movement greater than 0.1 dial divisions per minute.
4/ Coal caked and shrank away from retort wall; test discontinued.
5/ No resistance developed at normal rate of heating of 3° C. per minute.
6/ Coal swelled and stirring shaft froze at 430° C.; test discontinued.

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Expanding Properties of Coals During Carbonization

Another sole-heated expansion oven was constructed, and 62 tests have been made in it. A total of 169 tests was made in both ovens. Except for the method of applying pressure, the new oven was built to duplicate the older one as closely as possible so that a coal tested in either oven would give the same expansion within the limits of experimental error. The test results from the two ovens duplicate each other well.

A load of 2.2 pounds per square inch is applied on the coal charge during carbonization. In the older oven this pressure is maintained by manual adjustment of pressure in an oil gland controlling the pressure plate that rests on the coal charge. In the new oven a method maintaining automatically a constant pressure on the charge consists essentially of two hydraulic cylinders with pistons, one of small bore connected to another of larger bore so as to oppose each other. The piston of the small bore cylinder is appropriately weighted, and the liquid medium applies the same unit pressure to the large cylinder. The piston in this cylinder then continuously exerts the required pressure on the pressure plate contacting the coal. Figure 11 is a photograph of the new oven, showing the larger cylinder controlling the pressure plate.

The pressure plate of the new sole-heated oven was equipped with a cast iron base instead of transite, which was in use on the older oven. Comparison of results of tests using this plate and the older plate in the same oven indicated that the cast iron plate resting directly on the coal did not change the results significantly and gave more trouble-free operation, particularly with coals that became quite fluid; therefore, the transite part of the pressure plate of the older oven was replaced with cast iron.

The expanding properties of the coals and blends tested during the year are given in table 11. The State and bed for each coal is given to facilitate finding the complete description and chemical analyses given in table 8, wherein the coals are listed by States.

The Upper Freeport coals tested (470 from West Virginia, and 448, 458, and b480 from Pennsylvania) became very fluid during carbonization, and all but 448 were highly contracting. Blends of up to 30 percent Pocahontas No. 3 with coals 480 and b480 were still contracting enough to indicate they would be safe for use in commercial ovens, and coal 470 was so highly contracting that it would be expected to behave similarly. Blends of coal 458 with Pocahontas No. 3 were borderline coals that could be dangerous for most coke ovens. Coal 448 appears safe for industrial carbonization when used alone, but it was not tested in blends.

Of the other coals tested, the Pocahontas No. 3 is well-known to be a dangerously expanding coal when used alone. The tests indicated that the Lower Kittanning (443), the Sewell (465), the Sewanee (477), and the Beckley coals (449 and 450), when used alone, and a few of the blends would be potentially dangerous, particularly if coked at high speed in narrow ovens. A blend of two expanding coals, the Sewanee (477) and Pocahontas No. 3 (479), is commercially coked, with a carbonizing time of 30 hours, but oven damage is probably avoided because of the slow rate of carbonization in ovens that are wider than in many of the newer ovens.

A carbonizing-time study was made in the large vertical expansion oven with Pittsburgh-bed coal, Morgan mine No. 2, Perry County, W. Va., (446), under conditions simulating fast coking in very narrow slot ovens (8 inches wide). Wall temperatures used were higher than for those equivalent to flue temperatures 2,300° to 2,500° F. in commercial ovens. It was found that the carbonizing time in 8-inch ovens should be at least 6 hours to produce satisfactory furnace coke.

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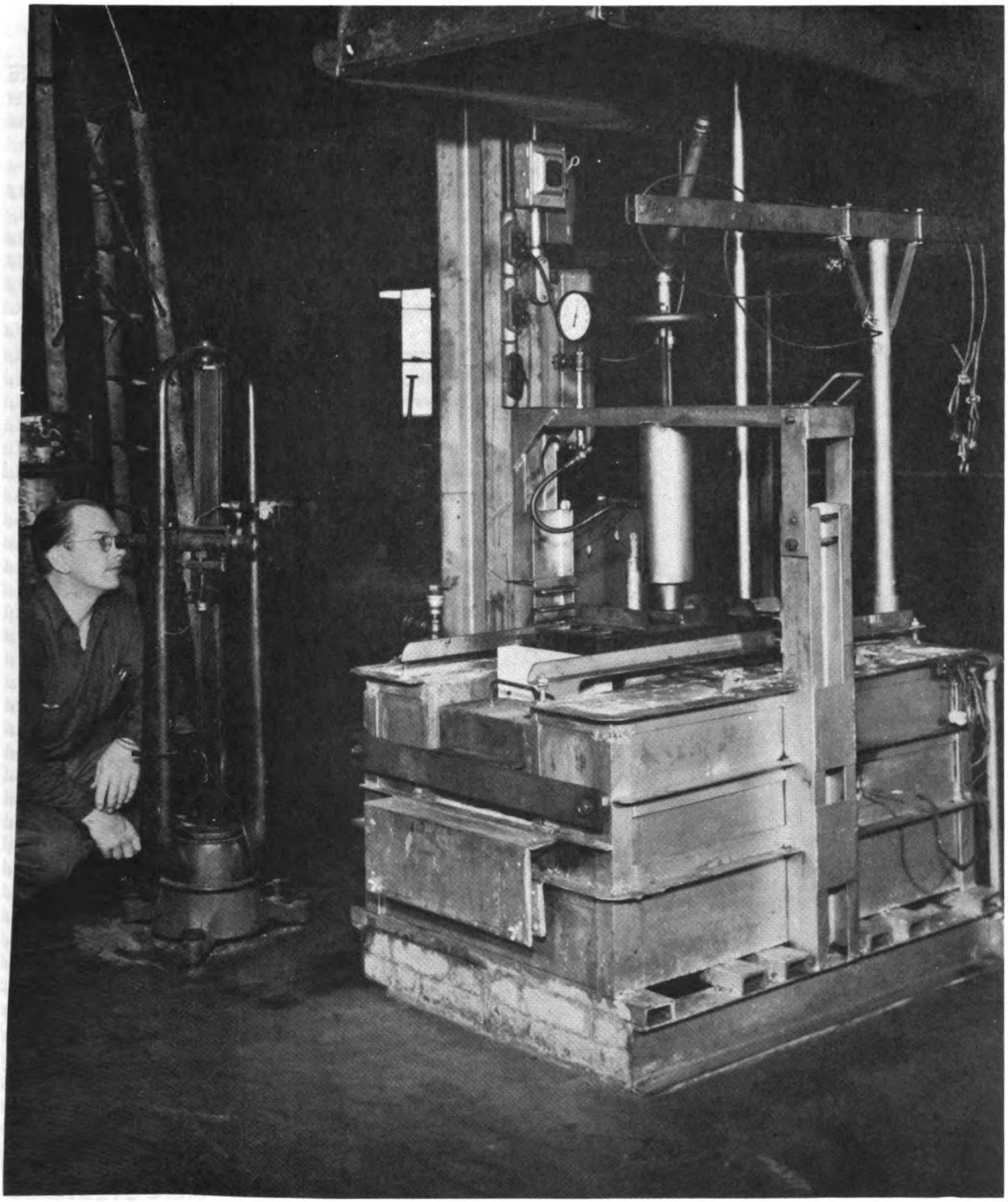

Figure 11. - New sole-heated expansion oven.

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TABLE 11. - Expanding properties of coal in the sole-heated oven

Coal No.	State	Bed	Test moisture, percent	Expansion, percent	
				At 55.5 pounds per cubic foot[1]	Dry, solid coal
s28	Pennsylvania	Pittsburgh	1.8	-17.3	24.0
h75	West Virginia	Pocahontas No. 3	2.7	+30.7	103.6
469[2,3]	Pennsylvania	Sewickley	2.9	-	-
469B[2]		Blend	2.6	-16.1	30.5
470[2]	West Virginia	Upper Freeport	3.3	-29.0	8.0
480[2]	Pennsylvania	do.	1.7	-21.3	21.4
b480[2]	do.	do.	1.9	-30.2	10.3
480A		Blend	1.7	-17.5	27.3
480B		do.	2.0	- 9.9	39.4
458	Pennsylvania	Upper Freeport	2.6	-14.8	30.6
458A		Blend	2.7	- .6	53.8
458B		do.	2.5	+ 4.5	61.2
448[3]	Pennsylvania	Upper Freeport	3.2	- 5.5	44.7
442	do.	Lower Freeport	2.5	-19.8	25.7
443	do.	Lower Kittanning	3.3	+14.4	74.8
442A		Blend	2.8	- 2.7	51.8
442B		do.	2.2	- 6.4	46.2
442C		do.	1.9	- 1.9	51.6
467	West Virginia	Winifrede	2.9	- 5.3	43.4
467A		Blend	2.4	+ 1.6	54.3
467B		do.	2.5	+ 3.1	56.8
467C		do.	2.9	- 1.5	49.2
467D		do.	2.9	+ 1.6	55.0
468	West Virginia	Winifrede	2.4	- 8.8	39.6
456	Tennessee	Jellico	2.7	- 7.2	42.3
456A		Blend	2.5	- 1.0	51.7
456B		do.	2.7	+ 1.0	53.9
476	Virginia	Tiller	2.5	- 1.1	47.0
465	West Virginia	Sewell	2.8	+ 6.5	58.0
465A		Blend	2.9	-11.3	37.5
465B		do.	3.1	- 8.8	41.7
465C		Blend	2.8	- 5.3	45.6
477	Tennessee	Sewanee	2.3	+ 5.8	63.3
478	Virginia	Clintwood	3.3	-17.7	27.1
479	West Virginia	Pocahontas No. 3	6.0	+17.7	86.7
477A		Blend	3.8	+ 8.2	70.2
477B		do.	2.6	- .4	56.2
477C		do.	2.3	+ 5.2	60.0
477D		do.	2.1	+ 1.2	55.5
449	West Virginia	Beckley	3.1	+ 7.5	70.5
449A		Blend	3.1	- 7.5	44.7
449B		do.	3.2	- 4.8	50.3
450	West Virginia	Beckley	1.6	+12.8	68.1
450A		Blend	2.9	- 3.9	49.0
450B		do.	2.7	- 1.4	53.7
475	Alabama	do.	2.5	- 8.9	39.6

1/ End-of-test contraction for contracting coals; maximum expansion for expanding coals.

2/ Single tests.

3/ Had contracted 18.2 percent when removed from oven in 3-1/2-hour test time; this is a highly contracting coal.

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Spontaneous Heating Tendencies of Coals and Refuse

The self-heating or spontaneous heating tendencies of five cleaned bituminous coals, five coal-refuse samples, and two lignites were determined under controlled oxidation in the adiabatic calorimeter. Table 12 shows the moisture condition of the samples as received, their analyses, and the self-heating rates at selected temperatures. Origin of the samples is given in table 8. The two Thick Freeport coals (444 and 445) that were very wet when received were air-dried before testing. The other coals, refuse, and lignites received in coarse sizes were crushed to 0-to 1/4-inch size, and all samples were dried in a rotary drum with nitrogen gas at 100° C. before testing in the calorimeter. The use of dry, sized coal under closely controlled conditions of oxygen supply and thermal equilibrium in the adiabatic calorimeter minimizes the effects of purely physical factors on the self-heating rate of the coal.

The Thick Freeport cleaned coal (444), with a high carbon content, self-heats at much faster rates at comparable temperatures than the Thick Freeport refuse coal (445), with a very high ash content, and the Lower Kittanning cleaned and refuse coals (451 and 452) behave in a similar manner. After 2 months of outdoor storage, the self-heating rates of the refuse coal (452-s2) increase and at comparable temperatures exceed those of the cleaned coal (451). These self-heating rates increase progressively as the time of outdoor storage of the refuse coal increase to 8 months. The surfaces of the particles of these two samples showed appreciable ferrous sulfate, some of which was lost before charging the dry samples in the adiabatic calorimeter. The increase in ferrous sulfate during 8 months of outdoor storage was from 0.46 to 1.56 percent (dry basis) for the refuse coals (452 and 452-s8). The accompanying decrease in pyritic sulfur in these two samples from 20.0 and 17.3 percent (dry basis) indicates that oxidation of pyrites was responsible in part for the high self-heating rates of the refuse coal (452-s8), which had been stored outdoors for 8 months.

The two cleaned Pittsburgh No. 8 coals (481 and 482) were relatively similar in chemical composition, but not in oxidizing properties. The higher rates found for coal 481 are in accord with the observed fact that this coal has heated and even ignited during shipment and storage, whereas coal 482 shows excellent storage properties. The cleaned Pittsburgh coal (s28), which was tested as a check with two other samples from the same mine previously tested, gave results that checked well for the three coals, showing that this coal is uniform in composition and has excellent storage properties. The high self-heating rates of the lignites indicate that spontaneous heating and ignition may be expected, unless proper precautions are taken in transporting and storing these coals.

Physical Properties of Coke: Size and Its Measurement

The adequacy of physical tests of coke for the purposes for which they are made has been a subject for discussion for many years. In 1944 the Bureau decided that data obtained from the testing procedures on samples of coke produced in commercial ovens would give information on (1) the degree of precision of tests commonly used in the industry, (2) the causes of the known poor reproducibility in several of the physical tests, and (3) possible improvements or modifications of existing physical testing procedures. Enough data have been collected on several aspects of coke testing to make presentation of data on the size of coke and its measurement worth while.67/

67/ Auvil, H. Stuart, and Gayle, John B., Physical Properties of Coke: Size and Its Measurements: Bureau of Mines Rept. of Investigations 4735, 1950, 27 pp.

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TABLE 12. - Analysis and self-heating rates of coals, coal refuses, and lignites

Coal No.	Condition of sample[1/]	Moisture as received, percent	Analysis, dry basis, percent					Self-heating rate in oxygen, °F./hour at given temperatures, °F.					
			Ash	H	C	Total S	Pyritic S	122	158	176	212	248	284
444.....	Cleaned, not dried, 0- to 1/4-in.	17.8	9.9	5.1	76.6	0.9	0.5	-	-	1.53	3.91	11.1	25.4
445.....	Refuse, not dried, 0- to 1/4-in.	16.3	65.6	1.7	22.0	8.7	8.2	-	-	.13	0.74	2.75	11.6
451.....	Cleaned, 3/8- to 1-1/4-in.	1.5	7.9	5.3	77.0	2.5	1.4	-	0.81	1.44	5.16	11.4	26.5
452.....	Refuse, 0- to 4-in.	1.5	43.9	2.5	34.0	21.0	20.0	-	.20	1.34	2.92	5.97	9.9
452-s2.....	Refuse, 0- to 1/4-in., stored outdoors 2 mo.	3.5	45.5	2.4	32.4	21.8	20.1	-	1.57	1.39	7.78	18.6	52.3
452-s4.....	Refuse, 0- to 1/4-in., stored outdoors 4 mo.	4.6	45.5	2.4	33.0	21.9	20.4	-	2.07	4.66	8.87	21.7	84.4
452-s8.....	Refuse, 0- to 1/4-in., stored outdoors 8 mo.	1.4	42.7	2.5	34.6	19.5	17.3	-	11.7	33.7	178.5	315.8	315.8
481.....	Cleaned and dried, 0- to 1/4-in.	5.0	7.9	5.1	75.1	2.9	1.7	-	-	5.22	10.5	29.5	44.2
482.....	Cleaned and dried, 0- to 1/4-in.	5.2	8.1	5.2	74.5	3.9	1.7	-	2.70	3.94	9.5	20.2	46.3
s28.....	Cleaned, 2- to 4-in. lump	1.3	5.3	-	-	-	-	-	-	2.30	2.90	4.85	Couple failed.
471.....	Raw lump lignite.....	37.0	14.1	4.2	61.3	0.7	-	12.8	23.0	28.9	76.5	109.0	79.1
473.....	Raw lump lignite.....	28.4	14.0	4.4	62.7	1.8	0.6	8.7	19.2	34.3	88.3	206.4	277.8

1/ After removing special moisture samples, coals 444 and 445 were air-dried before sampling; samples s28, 471, and 473 were crushed to nominal minus-1/4-in. size. All samples were dried in nitrogen at 100° C. in closed rotary-drum unit before charging to adiabatic calorimeter.

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For this study, samples of run-of-oven coke were taken at coke plants in the United States and Canada. Although sampling procedures were directed toward obtaining the highest possible degree of uniformity in the gross samples, it was found necessary to average the test results of a large number of 500-pound samples to reduce normal testing errors to a point where they were not appreciable fractions of the total variations found in a smoothly operating plant. For example, it was deduced that over 100,000 pounds of coke must be screened to determine if ordinary day-to-day variations in screen analysis have appreciable effects on the performance of a blast furnace. Reports on other aspects of testing the physical properties of coke are in preparation.

Methods of Making Coke

An introductory section was prepared for a chapter on coke in a recently published handbook on fuels and combustion.68/ Various types of coke and the equipment in which they are made were described. Cokes were classified according to use.

Low-Temperature Carbonization of Noncoking Coals

Small-scale laboratory low-temperature carbonization assays at 500° C. were completed on 55 samples of coal as part of the survey of properties of western coals. Samples ranging from lignite to high-volatile bituminous B coals were tested, and tar yields ranging from 14 to 55 gallons per ton were obtained. It was generally shown that the yield of tar from coal is approximately proportional to the volatile content.

A pilot plant for carbonizing noncoking coal in the entrained and fluidized state was operated successfully on several ranks of coal and lignite. The unit which was used (fig. 12), had a capacity of about 300 pounds of coal feed per hour. The pilot unit combines the new fluidized carbonizer and the fluidized dryer, which has been described in previous publications.69/ Carbonization is accomplished by passing the dried fine coal upward through an externally heated retort where the coal is fluidized and heated to about 900° F. All products move out the top of the retort. The coal is charged to the retort pneumatically with air, which combines with the char to liberate about half the heat required for carbonization inside of the retort. This method of processing and carbonizing fine material is applicable to all noncoking carbonaceous fuels, as shown by successful operation of the pilot plant on sawdust.

A larger pilot plant using the fluidized-bed drying and carbonizing technique, with a design capacity of 1 ton of coal per hour, was constructed at Denver before July 1, 1951. The same schematic arrangement was applied in the design of a large, commercial-scale pilot plant to treat approximately 25 tons of Texas lignite per hour. Such a unit is of commercial size, and large plants would consist of batteries of these units.

The object of this development work, which was conducted in cooperation with the Texas Power and Light Co., was to find an economical way to process Texas lignite to remove the valuable tars before burning the char for generation of electric power. Results from economic studies completed on large plants for processing 7,000 to 8,000 tons of lignite per day indicate that the char produced for combustion in the power plant will compete with natural gas, costing about 8 cents per thousand cubic feet when there is reasonable return from the sale of the tar.

68/ Newman, L. L., Methods of Making Coke: Fuels and Combustion Handbook (Allen J. Johnson, editor), McGraw-Hill Book Co., Inc., New York, N. Y., 1951, pp. 130-132.

69/ Parry, V. F., and Wagner, E. O., Drying Fine Coal in the Entrained and Fluidized State: Trans. Am. Inst. Min. and Met. Eng., vol. 187, 1950, pp. 974-982; Min. Eng., vol. 187, September 1950, pp. 974-982.

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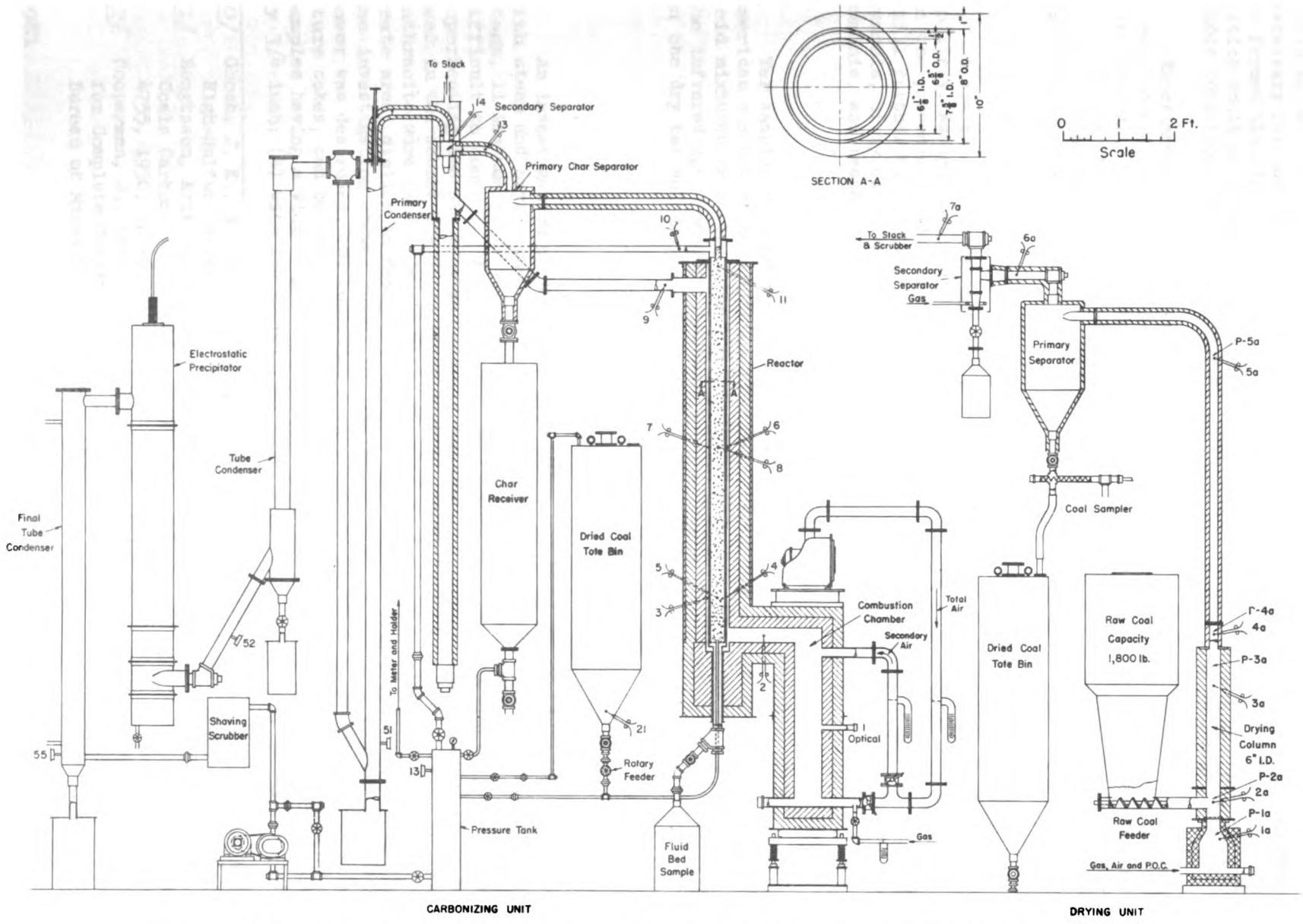

Figure 12. - Pilot plant for fluidized carbonization of noncoking coal; capacity, 250 to 300 pounds per hour.

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Low-temperature tars, containing up to 32 percent crude tar acids, are produced from typical Texas lignite by this process. These phenolic compounds and the higher-boiling homologues are potentially valuable in the plastics, wood-preservative, and tar-chemical industries. Gas produced in the process is used for supplying the heat necessary for carbonization. When carbonizing at 900° F., about 10 percent more gas is formed than is necessary for heating. In a large plant the temperature of carbonization would be adjusted to produce just enough gas to carry out the process. Under these conditions the overall thermal efficiency is approximately 88 percent.

Experimental work was conducted during the year on the briquetting of chars which were produced from the process described above to make domestic fuels, to substitute for charcoal. The char is very reactive; when bonded with starch it makes a smokeless briquet that burns almost like charcoal. Studies on the manufacture of substitutes for charcoal in Mexico, Cuba, and other foreign countries indicate that satisfactory fuels can be produced from various low-grade coals.

Desulfurization of Coal During Carbonization

A low-ash, good-coking, abnormally high-sulfur (7.5 percent) coal from Ledo Colliery, Assam, India, was effectively desulfurized by carbonization at 800° C. in the presence of added ammonia gas.[70] The desulfurizing effect was increased still further by addition of aluminum oxide to the coal before carbonization with ammonia, or by addition of sodium carbonate or lime before carbonization with ammonia, and leaching out the soluble sulfur from the cokes.

Phenol and Cresols in Coal Tar

Tar samples from BM-AGA carbonization tests at 800° and 900° C. of five American coals were investigated for their contents of phenol and cresols.[71] Tar-acid mixtures extracted from the dehydrated tars were fractionated and analyzed by the infrared spectrometer. Contents of phenol and each cresol isomer were reported on the dry tar and dry, ash-free coal.

GASIFICATION OF COAL AND LIGNITE

Lurgi Process

An investigation of the Lurgi process[72] for complete gasification of coals with steam and oxygen under pressure was carried out in three stages. In the first stage, 11 Alabama coals were tested in a 4-inch generator, which proved too small and difficult to control. A 6-inch batch generator, which was provided with facilities for superheating the steam-oxygen mixture and for synthesizing additional methane, was used in the second stage, in which Alabama and West Virginia coals, "Disco," and anthracite were used. In the third stage, a continuous generator of 1-square-foot grate area, similar in design to commercial German generators, was used. Results of the investigation showed that: (1) Caking coals could not be used unless their coking power was destroyed; (2) noncaking fuels, such as anthracite and low- and high-temperature cokes, can be completely gasified; (3) although it was possible to gasify samples having a wide range of sizes, the optimum size range of the generator was 1/8- by 3/4-inch; (4) with the proper fuel size, a gas-making capacity as high as 9,000

70/ Ghosh, J. K., and Brewer, R. E., Desulfurization of Coal During Carbonization, High-Sulfur Indian Coal: Ind. Eng. Chem., vol. 42, 1950, pp. 1550-1558.

71/ Bengtsson, Erik, (revised by R. E. Brewer), Phenol and Cresols in Coal Tar from Coals Carbonized at 800° and 900° C.: Bureau of Mines Rept. of Investigations 4755, 1950, 10 pp.

72/ Cooperman, J., Davis, J. D., Seymour W., and Ruckes, W. L., Lurgi Process: Use for Complete Gasification of Coals with Steam and Oxygen under Pressure: Bureau of Mines Bull. 498, 1951, 38 pp.

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cubic feet per hour per square foot of gasification area was obtained; (5) the heating value of the carbon dioxide-free gas from bituminous char was about 410 B.t.u. per cubic foot, which dropped slightly at high capacities; (6) using the British nickel methanization catalyst, gas (free of carbon dioxide) of about 950 B.t.u. per cubic foot was synthesized from the Lurgi gas; (7) the soda-iron oxide catalyst used to convert and remove organic sulfur required 10 to 15 times the volume of the nickel catalyst because of its slow action.

Lignite Gasification

Investigation of the continuous gasification of lignite in the externally heated annular retort at the lignite pilot plant at Grand Forks, N. Dak., was continued during the year. Results of three tests conducted in the pilot plant are given in table 13.

TABLE 13. - Results of gasification tests of lignite, 1951

Test No.	Gasification, hours	Natural lignite used, pounds	Gas made, cu. ft. (S.G.C.)1/
14.....	802	370,440	8,220,423
15.....	466	257,908	4,872,542
16.....	637	306,228	6,965,045
Total.....	1,905	934,576	20,058,010

1/ S. G. C., standard gas condition; saturated gas at 60° F. and 30 inches of mercury.

The alloy retort tube is the heart of the gasification unit. The present 310 alloy tube is the fourth in a series that has been tested and is by far the most satisfactory tube. It has been in operation 3,767 hours, or approximately as long as the other three tubes combined, and has handled more lignite and has produced considerably more gas than the other three tubes. At the end of the year the tube was still in entirely satisfactory condition for further operation, and there was no indication as to its probable minimum useful life.

The gasification of lignite in this pilot plant, the design and operating problems of the plant, and the analyses of the gases produced under various operating conditions have been published.73/

A review74/ was published of the gas-composition data taken in the various test runs and worked out as a basis for correlating the results. The composition of the gases, the H_2/CO ratio, the heating value of the gas, and the gas production per unit weight of carbon gasified are shown to be related to the fraction of undecomposed steam present in the product gas.

Underground Gasification

Interest in underground gasification of coal has been widespread. The possible advantages of underground gasification are summarized as follows: A reduction in invested capital costs; the recovery of the energy of the coal in an easily used

73/ Holtz, J. C., Gasification of Lignite in the Commercial-Scale Pilot Plant at Grand Forks: Proc. of the North Dakota Academy of Science, vol. 4, November 1950, pp. 39-42.

74/ Chetrick, M. H., Undecomposed Steam in Lignite Gasification: Bureau of Mines Rept. of Investigations 4738, 1950, 11 pp.

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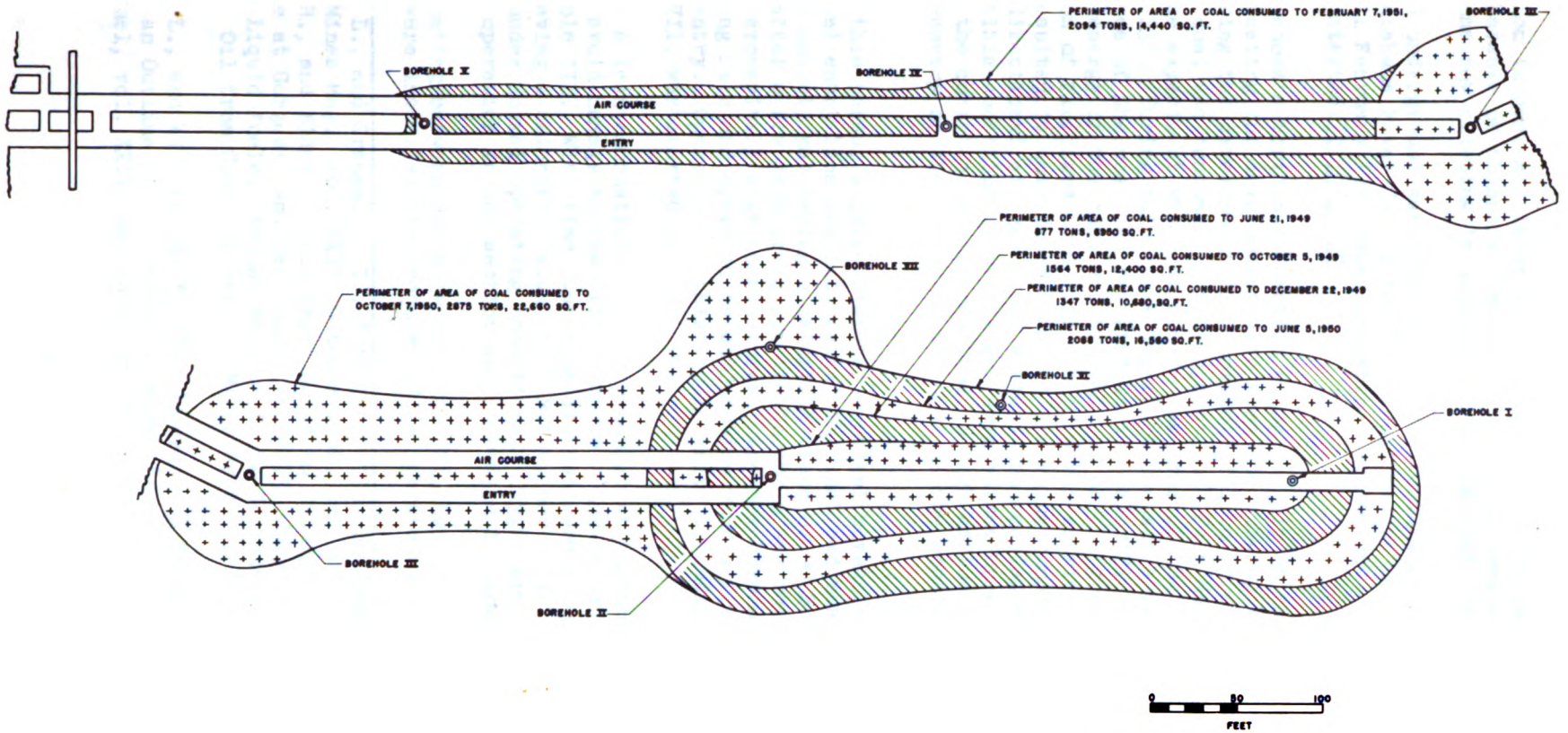
PERIMETER OF AREA OF COAL CONSUMED TO FEBRUARY 7, 1951,
2094 TONS, 14,440 SQ. FT.
BOREHOLE IV
BOREHOLE V
BOREHOLE III
AIR COURSE
ENTRY
PERIMETER OF AREA OF COAL CONSUMED TO JUNE 21, 1949
877 TONS, 6950 SQ. FT.
PERIMETER OF AREA OF COAL CONSUMED TO OCTOBER 5, 1949
1564 TONS, 12,400 SQ. FT.
PERIMETER OF AREA OF COAL CONSUMED TO DECEMBER 22, 1949
1347 TONS, 10,680, SQ. FT.
PERIMETER OF AREA OF COAL CONSUMED TO JUNE 5, 1950
2088 TONS, 16,560 SQ. FT.
PERIMETER OF AREA OF COAL CONSUMED TO
OCTOBER 7, 1950, 2875 TONS, 22,660 SQ. FT.
BOREHOLE VII
BOREHOLE VI
BOREHOLE I
AIR COURSE
ENTRY
BOREHOLE II
BOREHOLE VIII
0
50
100
FEET

Figure 13. - Coal consumed and area of burned-out region, underground gasification test, Gorgas, Ala.

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form; a reduction in over-all labor requirements; the utilization of coal deposits now unminable because of thinness, impurities, or other reasons; and the elimination of the conditions now existing in underground mining that are hazardous to personnel.

The second underground gasification project conducted jointly by the Bureau of Mines and the Alabama Power Co. in the Pratt coal bed at Gorgas, Ala., was continued under fire until February 1951, when operations ceased and flooding of the system with water was started.75/76/77/78/

Figure 13 shows a horizontal section along the dip of the bed, the upper part of the figure indicating the development work from the outcrop to borehole III and the lower part showing the development from borehole III to borehole I, where the major portion of the coal was consumed in this operation. From March 1949 through May 1950 operation of the experiment was confined to the area of the coal bed between boreholes I and II (fig. 13). During this phase imperfect contact between the injected air and the exposed faces of the coal bed resulted in low calorific values in the effluent gases, and the energy of the coal was brought out of the ground largely as sensible heat. Injection of fluidized sand into the prepared entries and into burned areas of the coal bed resulted in some improvement in operating conditions. The use of long periods of unidirectional air-gas flows (whereby the gas issued at high temperatures, and the combustible constituents of the gas were burned upon contact with unreacted air in or near the outlet borehole) resulted in high effluent gas temperatures and aided in the recovery of the energy of the coal as the sensible heat of the gaseous products.

Following this phase, a large-diameter vertical borehole (VI) was drilled away from the original entry line and intersected the coal bed near the reacting face. When used as an outlet, gas having a calorific value of 90 B.t.u. per cubic foot was produced, but after a few days operation the borehole was rendered useless by the development of excessively high temperatures. Another borehole (VII) was constructed near the reacting face and operated in conjunction with a fresh section of the original prepared entry. Operation of this second unit, including the area between boreholes III and VII, was started in June 1950.

Initially, a lean combustible gas was produced at borehole VII. The air input was limited to avoid damage to the exit borehole. Low-calorific value gas was produced at borehole III. With time, gas equality declined at each outlet; after 4 months a gas having a calorific value of 20 B.t.u. per cubic foot was produced at either outlet under normal operating conditions. Oxygen appeared in the gas a week after starting operation in the section and increased in amount with time.

While operating between boreholes III and VII, two aircraft-type turbosupercharger units were installed at borehole III to operate by hot gas from the system,

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75/ Elder, J. L., and Graham, H. G., Experimentation on Underground Gasification of Coal: Mines Mag., vol. XXXX, October 1950, pp. 107-111, 117, 134.

76/ Fies, M. H., and Elder, J. L., Experiments on Underground Gasification of Coal Continue at Gorgas: Am. Gas Jour., vol. 173, July 1950, pp. 15-18.

77/ Synthetic Liquid Fuels. Annual Report of the Secretary of the Interior for 1950. Part I. Oil from Coal: Bureau of Mines Rept. of Investigations 4770, 1951, 74 pp.

78/ Elder, J. L., and Wilkins, E. T., The Underground Gasification of Coal in the U.S.A., an Outline of the Bureau of Mines Experiments at Gorgas, Ala., Jour. Inst. Fuel, vol. XXIV, May 1951, pp. 94-99.

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as shown in figure 14. The compressed air from the superchargers was added to that from the reciprocating compressor and injected underground. Early in these tests, the gas ignited underground as a result of the combustible constituents uniting with the free oxygen. Gas-discharge temperatures ranged from 1,000° to 1,400° F. Pressures became stabilized at 18 p.s.i.g. air-inlet pressure and 12 p.s.i.g. gas-outlet pressure. Under these conditions the oxygen content of the gas decreased to zero, and the turbine discharges burned freely. No unreacted air reached the outlet borehole. The operation of the turbines qualitatively demonstrates a means of recovering the sensible heat of the gases while simultaneously creating more favorable conditions for gasification.

The last operating phase of the experimental installation was to determine if a longer coal face exposed to gas flow would be advantageous. This operation was between boreholes III and V, which provided 600 feet of path, initially bordered by four coal faces. Free oxygen appeared after several cycles, and the calorific value of the gas was low. Material balances show that the amount of coal consumed was equivalent to the entire pillar between the entries plus 10 feet off the sides of the entry in the solid coal. The operating characteristics of the section were similar to those encountered previously. The increased face area at first exposed in this operation increased the rate of coal consumption but had little effect on gas quality. A very long cycle of unidirection flow resulted in high gas temperatures, with little change in analysis, and greatly increased the fraction of the energy of the coal appearing as sensible heat in the products.

Several periods of low input air rates were made to determine this effect. The generation of steam in both the old and contemporary operating areas caused the results to be inconclusive; the proportion of water vapor in the product gas was high. Lowering the air input rates led to initial increased calorific value of the gas, which was due to the increase in the reaction between steam and coke. Prolonged operation at low air rates was characterized by decreasing calorific value; this was probably due to general cooling of the reacting faces.

In February 1951 operations ceased and flooding of the system with water was started. Cooling has progressed satisfactorily to the present time. The operation at Gorgas has demonstrated that the combustion of coal in place can be extended over a wide area from a single initial passageway in the coal bed, as shown in figure 13. It has been possible to advance a burning face still further into the solid coal by drilling boreholes to intersect the face. The low calorific value of the gas produced was due to incomplete contact between air and coal caused by voids formed in or above the original entries and in the burned out area. These voids allowed the air to bypass the coal faces. Efforts to fill these voids with dry, fluidized solids were only partly successful. Favorable conditions for gasification were obtained when the gas outlet was located very near an incandescent carbon face. The quality of the gas decreased as the reacting face receded from the borehole; increasing pressure, temperature, and gas velocity near the outlet, in the presence of hot carbon, resulted in improved quality of gas.

It was possible to bring a large proportion of the energy of the coal to the surface as sensible heat when desired. An installation, whereby the hot gas is expanded through a turbine, would require gas-tight strata and minimum pressure drop through the system, but many coal beds retain natural gas under pressure, and these beds may fulfill requirements for gas turbine operation.

The most urgent problem in underground gasification is control of the contact between air and coal. With contact assured, gasification will result, and it will be

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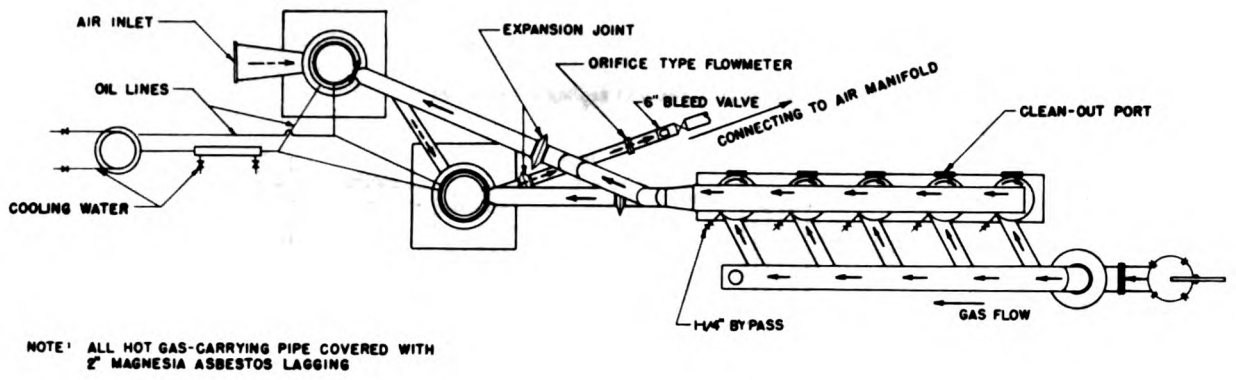

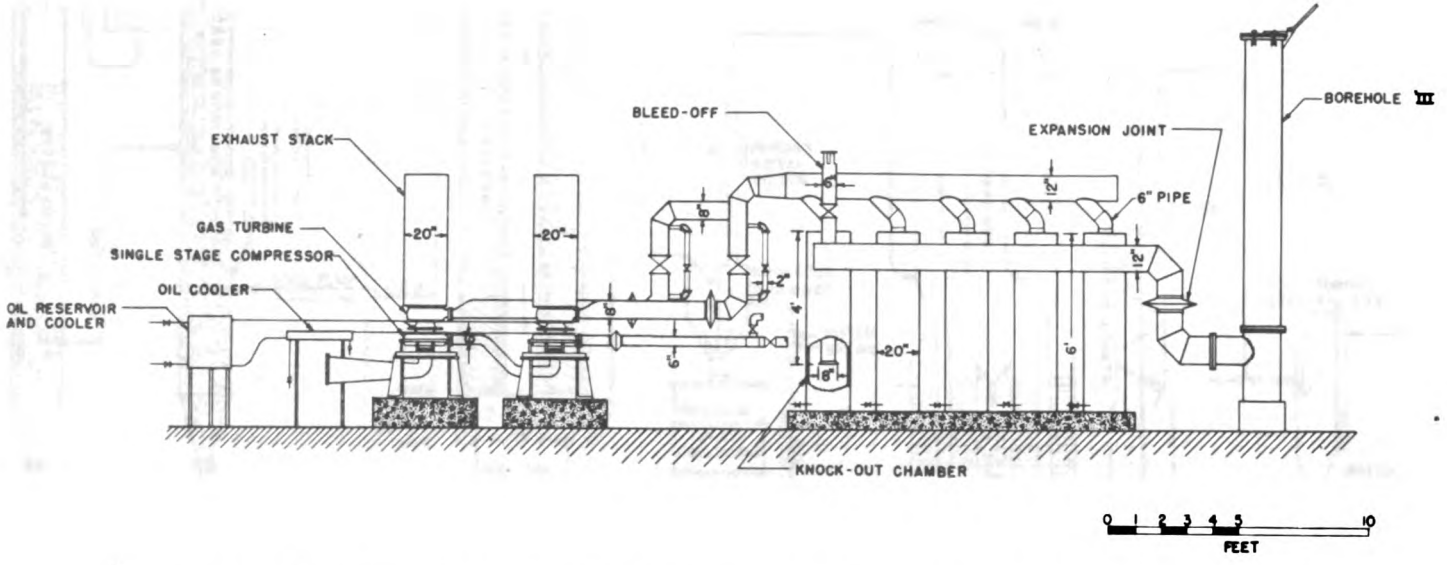

Figure 14. - Gas turbine installation, underground gasification test, Gorgas, Ala.

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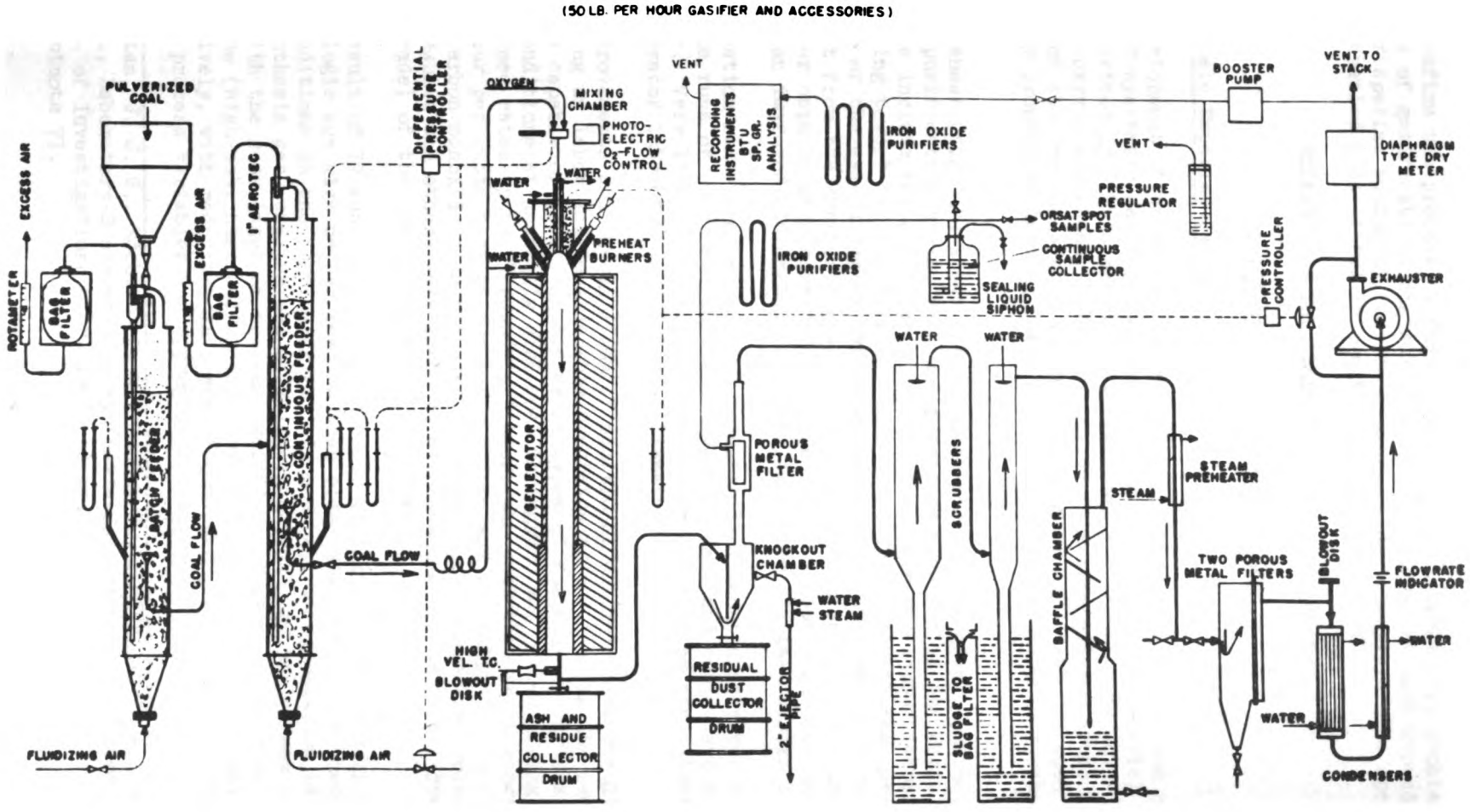

Figure 15. - Flow diagram of apparatus for testing fuels for synthesis-gas production.

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possible to refine the process by further development. Subsidiary problems, such as optimum rates of gasification, control of leakage, methods of underground development, and design of special equipment, can be attacked simultaneously. The operation at Gorgas has revealed much useful information on these problems.

RESEARCH ON THE PRODUCTION OF SYNTHESIS GAS

Gasification of Pulverized Coal

Laboratory-Scale Experimentation

The development of an easily controlled laboratory process for the continuous production of synthesis gas from powdered coal at ordinary (atmospheric) pressures has been described.79/ This method, powdered-coal gasification by down-draft entrainment in oxygen and steam, has been considerably standardized after some further development of the feeding system and purification train80/ and was used to carry out a preliminary program of testing fuels for their relative value in synthesis-gas production.

A flow sheet (fig. 15) shows the 50-pound-per-hour gasifier, pneumatic feeding system, and purification train, with modifications made during the year. These modifications include the substitution of a much larger continuous feeder (10-inch i.d. fluidizing tower) for the former 4-inch diameter vessel and the use of a single batch feeder of the same diameter, as shown in the flow diagram. The dry method of removing dust from the synthesis gas was replaced by two efficient scrubbers and a baffle chamber capable of recovering the bulk of the carbonaceous residual dust, followed by an improved micrometallic filter to remove the last traces of residue.

Gasification runs were, as a rule, voluntarily discontinued after 4 to 5 hours, although some runs of 23 hours' duration have been made and could have been continued indefinitely. Very little slag formation was ever observed on the 7-foot silicon carbide generator tube of 6-inch internal diameter.

The improvements and installation of additional controls have resulted in a greater saving of labor and considerable precision in duplicating the results. The apparatus is capable of gasifying different types of fuels under variously chosen operating conditions at unusually high coal-throughput and corresponding gas-output rates. At feed rates up to 55 pounds per hour, the coal throughput ranged up to 42 pounds an hour per cubic foot of generator volume and the output up to 920 cubic feet an hour of carbon monoxide plus hydrogen per cubic foot of gasifier volume. These operating results and others were found to be functions of the rank (type) and composition (grade) of the coal gasified.

As a result of 79 runs made, it is clearly indicated that low-rank coals of younger geologic age give better results than older high-rank coals when tested under the same conditions in the same gasifier. In general, the completion of gasification, yield of synthesis gas, and thermal efficiency decrease and the oxygen requirement increases with the rank (age) of the coal gasified. However, less-reactive, high-rank and low-grade (high-ash, high-sulfur and low-ash fusion point) coals may also be gasified effectively, with reduction in oxygen requirement, by a suitable choice and combination of process variables and appropriate changes in the generator design. The

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79/ Sebastian, J. J. S., Edeburn, P. W., Bonar, F., Bonifield, L. W., and Schmidt, L. D., Laboratory-Scale Work on Synthesis-Gas Production: Bureau of Mines Rept. of Investigations 4742, 1951, 41 pp.

80/ See footnote 77.

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modifications required include reduction of heat losses, provision for longer contact time and increased mass transfer rate (diffusion), and suitable refractory lining to withstand higher generator temperatures. Thermodynamic and stoichiometric calculations and translations of the results to operations on large commercial scale confirmed these conclusions derived from experimental data.

Gasification by the vortex Principle

A progress report[81] presented the preliminary results of an investigation on the production of synthesis gas from low-cost pulverized coal and steam-oxygen mixtures in a vortex reactor. The basic principles of the reactor design and an illustrative example of their application were given. A method of calculating, a priori, the composition of the make gas for any set of feed conditions was presented and has been checked experimentally.

Subsequent work has shown the performance of the vortex to be vastly improved by changing the geometry of the vortex and by introducing the coal horizontally into the reactor, so that it is thrown into the rotating gas stream in an umbrella-like pattern.

Atmospheric Pressure Pilot-Plant Gasifier

During the year test work was continued on the 500-pound-per-hour atmospheric pressure unit previously described.[82][83] The generator was rebuilt to permit continuous slag tapping, the generator-base construction being modified from time to time to improve the slag tapping, and 10 tsst runs were made. Effective slag tapping was attained in these tests with oxygen consumption of approximately 320 cubic feet per MCF of CO + H_2 produced, using steam at 1,000° F. In the early tests it has been indicated that extremely fine pulverization of the coal (90 percent through a 200-mesh screen) was probably not required for good gasification results. In these tests, coarser fuel was fed (as coarse as could be handled in the pneumatic feeder) in some of the runs, and the results indicated that the percent of gasification was satisfactory. After run 42 it was apparent that the limit of profitable experimental work had been reached, using the original generator.

Under a cooperative agreement with the Babcock & Wilcox Co., a new generator was designed. The shell was fabricated in its shops, and the construction on the site, including installation of refractories, was done by Bureau personnel. This new unit will make possible further studies of methods of slag removal and reactant feeding. The dust-removal train, previously used, functioned very well, and no major changes have been made in it.

A patent has been issued[84] covering gasification of pulverized coal with oxygen and steam. Pulverized coal entrained in a stream of oxygen is admitted near the base of a generator through diametrically opposed ports so that the flames impinge on each

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81/ Perry, H., Corey, R. C. and Elliott, M. A., Continuous Gasification of Pulverized Coal with Oxygen and Steam by the Vortex Principle: Trans. Am. Soc. Mech. Eng., vol. 72, No. 5, July 1950, pp. 599-610.

82/ Strimbeck, G. R., Holden, J. H., Rockenbach, L. P., Cordiner, J. B., Jr., and Schmidt, L. D., Pilot-Plant Gasification of Pulverized Coal with Oxygen and Highly Superheated Steam: Bureau of Mines Rept. of Investigations 4733, 1950, 41 pages. Abs. in Proc. Am. Gas Assoc., 1950, pp. 501-502.

83/ See footnote 77.

84/ Newman, L. L. and Donovan, J. T., Apparatus and Method of Gasifying Carbonaceous Material: U. S. Patent 2,516,141, July 25, 1950.

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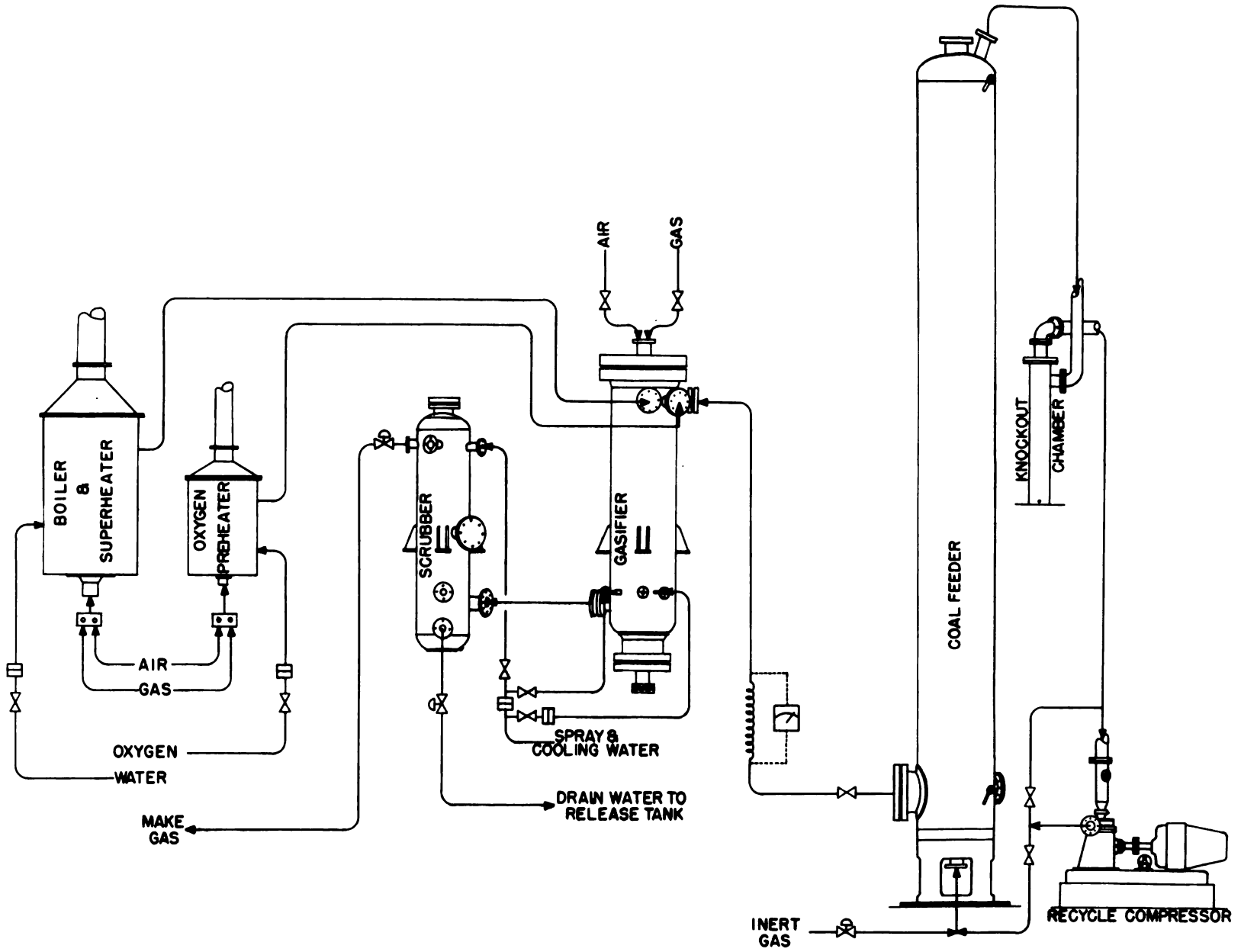

Figure 16. - Simplified flow sheet for pressure gasification.

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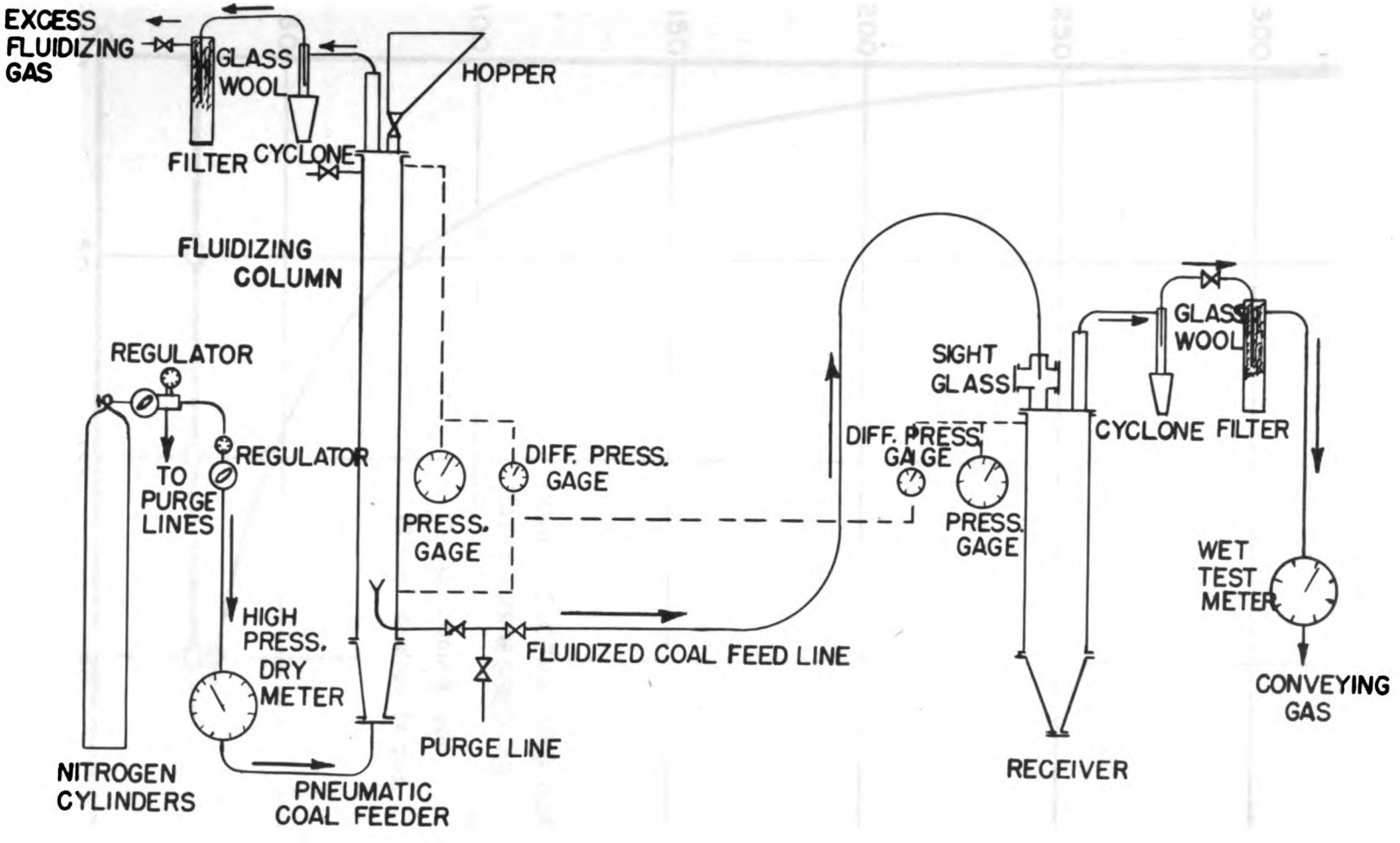

Figure 17. - Apparatus for study of transporting and feeding powdered coal under pressure.

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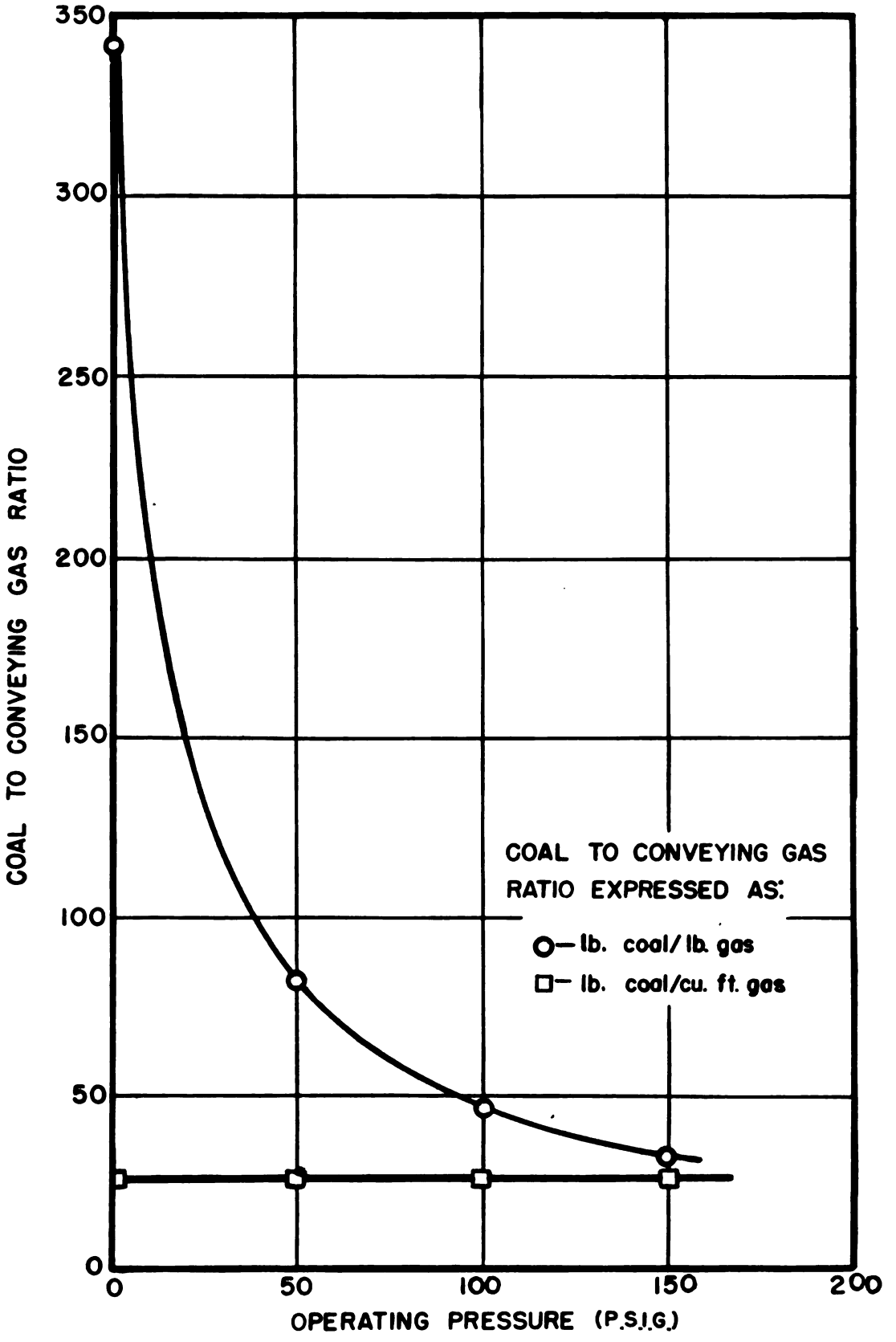

Figure 18. - Effect of operating pressure on the amount of coal conveyed by air or other gases.

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other with considerable turbulence. Steam is admitted below the oxygen-coal ports through tangential ports. The process can be used either at atmospheric or high pressures.

High-Pressure Pilot-Plant Gasifier

The construction of the high-pressure gasification pilot plant[85] was finished, and test runs were started in June 1951. To date these tests have been of short duration to check the mechanical operation of the various units and to train operating personnel. The performance of the various units has been satisfactory and indicates the correctness of the original design. Figure 16 gives a flow diagram for this high-pressure pilot plant.

Testing an experimental, coke shaft filter has continued. Excellent results in removing very fine dusts have been obtained in the bench-scale apparatus, with low-pressure losses.

Pneumatic Transportation and Feeding of Pulverized Coal

As the high cost of compression of the synthesis gas, before purification and synthesis to liquid fuels, favors gasification at elevated pressures, a method was developed for the continuous pneumatic transportation and charging of powdered coal into a pressurized gas generator.[86] Experimental work on a laboratory scale,[87] under pressures up to 150 pounds per square inch, proved that powdered coal can be transported pneumatically in a dense phase (26 pounds per cubic foot of space in the conveying pipeline) from a fluidized bed and discharged at a constant rate. A flow diagram (fig. 17) shows the apparatus constructed for studying the continuous conveying and feeding of finely powdered coal at superatmospheric pressures.

The results of the experimental work showed that, with other conditions equal, the discharge rate is solely a function of the pressure differential across a conveying tube of given dimensions. It was shown further (fig. 18) that, regardless of the operating pressure, the amount of coal conveyed per unit volume of gas, measured at the existing pressure, is practically constant, but the weight of coal carried per unit weight of conveying gas rapidly decreases with increasing operating pressures. The design information published on pressurized dense phase feeders has not been available heretofore.

Synthesis-Gas Purification

Of the organic sulfur compounds which may be present in synthesis gas, thiophene is known to be the most resistant to thermal and catalytic treatment. An analytical method using isatin was developed for determining thiophene in gas. With this method, as little as 0.0001 grain of thiophene sulfur per 54.5 ml. of test solution could be detected.[88] Tests on the gas produced in the first two atmospheric pressure gasifiers showed that no thiophene sulfur was present.

85/ Synthetic Liquid Fuels. Annual Report of the Secretary of the Interior for 1950. Part I. Oil from Coal: Bureau of Mines Rept. of Investigations 4770, 1951, 74 pp.

86/ Holden, J. H., and Albright, C. W., The Morgantown Fluidized Feeder: Proc. Am. Gas Assoc., 1950, pp. 493-500.

87/ Barker, K. R., Sebastian, J.J.S., Schmidt, L. D., and Simons, H. P., Pressure Feeder for Powdered Coal or Other Finely Divided Solids: Ind. Eng. Chem., vol. 43, May 1951, pp. 1204-1209.

88/ Wainwright, H. W., and Lambert, G. I., A Colorimetric Method for the Determination of Thiophene in Synthesis Gas: Bureau of Mines Rept. of Investigations 4753, 1950, 11 pages.

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A method was developed for continuously determining low concentrations of dust in gas by means of an instrument which compares, by means of photoelectric cells, the intensity of a light beam passing through the dust-laden gas and a similar beam through clean air.89/ This instrument had a sensitivity of 0.75 grain per 100 cubic feet. The gas-purification research program at Morgantown always contemplated the necessity of including sulfur recovery as a feature of the gas-purification system in any large gas-synthesis plant.90/ The first gas-purification pilot plant runs were made at 300 p.s.i.g., using 20, 30, 35, and 50 percent aqueous solutions of triethanolamine (TEA) as a selective absorbent for hydrogen sulfide. Data indicate that a 30 to 35 percent solution is the optimum concentration, if both selectivity and economy are considered. Using 35 percent TEA and a raw gas containing about 350 grains of hydrogen sulfide per 100 s.c.f. and 15 percent carbon dioxide, an acid gas containing about 10 percent hydrogen sulfide is obtained. Such an acid gas can be used for sulfur recovery, but not as economically as an acid gas having a higher hydrogen-sulfide content.

Slightly better selectivity is obtained with 20 percent TEA solution, but steam consumption for reactivation is greater, owing to higher solution rates. As a result of the greater solubility of carbon dioxide, a 50 percent TEA solution not only produces an acid gas of lower hydrogen-sulfide content but it also requires high solution rates to effect hydrogen-sulfide removal from the gas.

In cooperation with the Southern Natural Gas Co., investigations were made at 300 p.s.i.g. on the simultaneous removal of hydrogen sulfide and carbon dioxide from gases employing various concentrations of diethanolamine (DEA). Both TEA and DEA reduced considerably the organic sulfur content of the raw synthesis gas. Analyses of the lean amine solutions showed a gradual build-up of sulfur compounds, indicating that the compounds formed by the amines and the organic sulfur do not break down under the ordinary operating conditions of the reactivator. The quantity of steam needed to reactivate fouled TEA and DEA solutions increased as the reactivator pressure was increased. There was considerable difficulty in operating the pilot plant owing to foaming of the amine solutions. Initial additions of antifoam Alkaterge C agent were successful in reducing the foaming in the pilot-plant absorber, but further additions were less effective.

The potassium-phosphate process for the selective removal of hydrogen sulfide has also been investigated on a pilot-plant scale. Data, thus far, indicate that, with identical gas, this process will give an acid gas containing about twice as much hydrogen sulfide as that obtained with triethanolamine. It is generally thought that the phosphate process is costly because excessive steam is consumed during reactivation, but pilot-plant results have shown this process to be no more expensive than the amine process.

In addition to the removal of hydrogen sulfide and carbon dioxide, pilot-plant runs have been made in which the organic sulfur has been removed from raw synthesis gas with "GW" activated carbon. At present, the activated carbon process for removing organic sulfur appears to be the most economical. If the raw gas entering the carbon purifiers contains about 10 percent carbon dioxide, it is known from laboratory tests that this amount of carbon dioxide in the gas reduces the carbon's capacity for sulfur

89/ Stone, D. E., Kane, L. J., Corrigan, T. E., Wainwright, H. W., and Seibert, C. B., Investigation of a Photoelectric Device for the Determination of Low Concentrations of Dust: Bureau of Mines Rept. of Investigations 4782, 1951, 6 pages.

90/ Sands, A. E., and Schmidt, L. D., Recovery of Sulfur from Synthesis Gas: Ind. Eng. Chem., vol. 42, November 1950, pp. 2277-2287.

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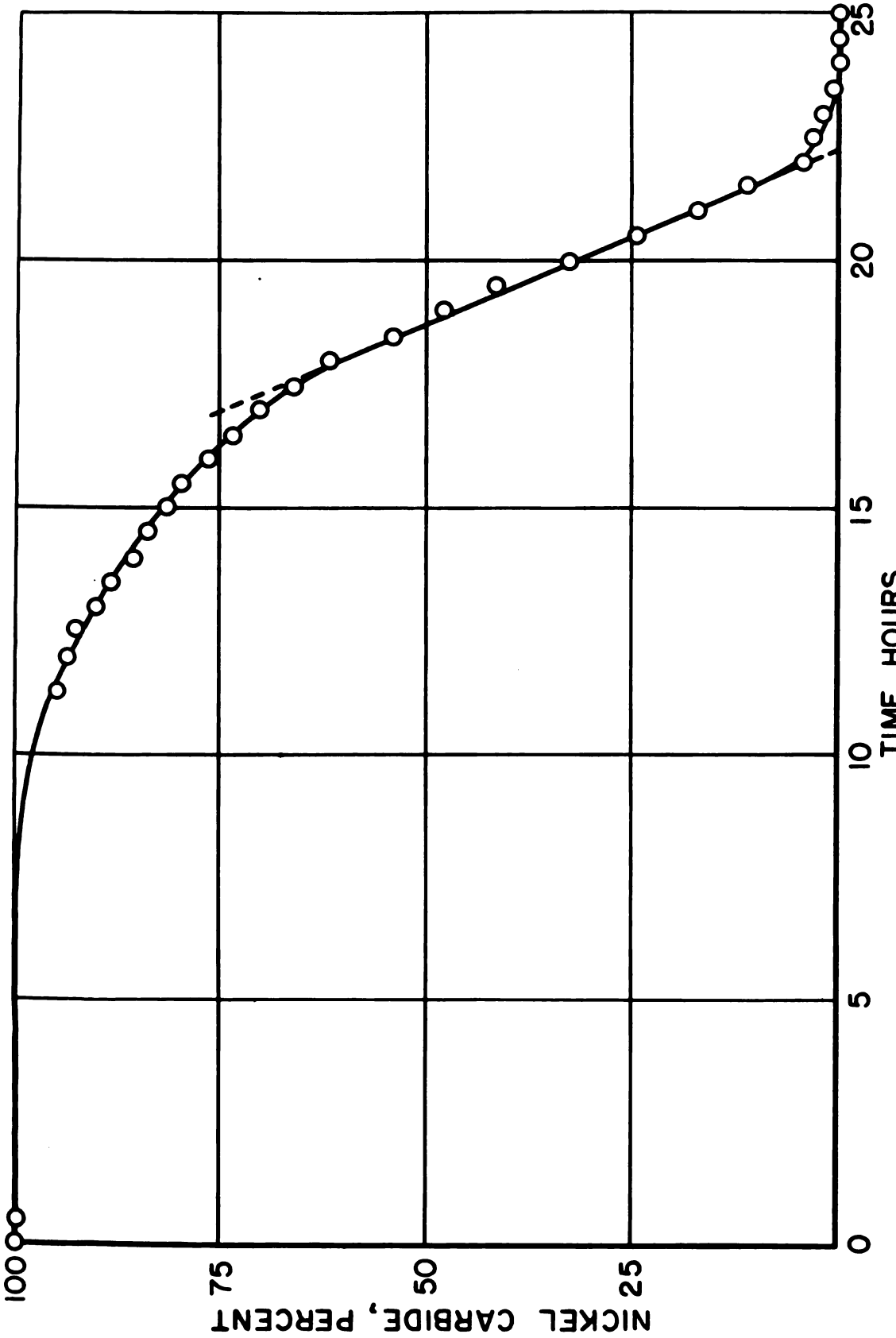

Figure 19. - Isothermal decomposition of fully carburized Ni_3C at 323° C.

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about one-half.91/ If the carbon dioxide is to be removed before the Fischer-Tropsch synthesis, it should be removed before the carbon adsorption step, which would make the carbon process even more attractive.

By first removing selectively the bulk of the hydrogen sulfide from raw synthesis gas, then removing the residual hydrogen sulfide with iron oxide, and finally removing all organic sulfur compounds with activated carbon, it is possible to reduce the sulfur content of synthesis gas to less than 0.1 grain per 100 s.c.f. During most of the pilot plant runs, the sulfur content of the purified synthesis gas was less than 0.05 grain per 100 s.c.f.

SYNTHETIC LIQUID FUELS BY GAS SYNTHESIS: RESEARCH AND DEVELOPMENT

Mechanism of the Synthesis

A generalized relationship was developed between reaction rates and the thermodynamics of a system near equilibrium.92/ Equations were derived by which the magnitude of the forward (or backward) reaction rate at equilibrium may be determined from the net reaction rate close to equilibrium. These equations may be used in the study of catalytic processes, such as the water-gas shift reaction, important in the Fischer-Tropsch synthesis, and in the study of homogeneous gas reactions, such as those which occur in rocket nozzles.

For an understanding of the mechanism of the Fischer-Tropsch synthesis and for an estimate of the relative amounts of the products, the product distribution must be known or predictable. The products of the synthesis are not in thermodynamic equilibrium with respect to the types of molecules, structures of the carbon chains, and possibly carbon number distribution. Hence the nature of the products is at least partly determined by the reaction mechanism. Simple rules for stepwise carbon addition predict satisfactorily the experimentally determined hydrocarbon isomer and carbon number distributions in products obtained with cobalt and iron catalysts.93/ According to this mechanism, addition can occur only at end or penultimate carbon atoms of the longest carbon chain and is not possible on a penultimate carbon already attached to three carbon atoms.

Studies of Catalysts

Metallic Carbides

A series of isothermal decompositions of nickel carbide was made in the study of carbon deposition on catalysts.94/ Samples of pure nickel were carburized with carbon monoxide to form nickel carbide, Ni_3C. This was heated at constant temperature, and the course of the decomposition was followed by measuring the increase in ferromagnetism. An induction period preceded the decomposition as shown in figure 19.

91/ Sands, A. E., Wainwright, H. W., and Egleson, G. C., Organic Sulfur in Synthesis Gas: Occurrence, Determination, and Removal: Bureau of Mines Rept. of Investigations 4699, 1950, 51 pages; Proc. Am. Gas Assoc., 1950, pp. 564-602.

92/ Manes, M., Hofer, L.J.E., and Weller, S., Classical Thermodynamics and Reaction Rates Close to Equilibrium: Jour. Chem. Phys., vol. 18, October 1950, pp. 1355-1361.

93/ Anderson, R. B., Friedel, R. A., and Storch, H. H., Fischer-Tropsch Reaction Mechanism Involving Stepwise Growth of Carbon Chain: Jour. Chem. Phys., vol. 19, March 1951, pp. 313-319.

94/ Hofer, L.J.E., Cohn, E. M., and Peebles, W. C., Isothermal Decomposition of Nickel Carbide: Jour. Phys. Colloid Chem., vol. 54, November 1950, pp. 1161-1169.

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There was no detectable change in the structure of the carbide during the induction period, that is, it decomposed into metallic nickel and carbon without the formation of an intermediate carbide. For the period of the decomposition indicated by the linear portion of the curve in figure 19, the reaction was of zero order. Shortening the induction period by temporary overheating did not affect the zero-order rate of the decomposition, except when the normal induction period was completely suppressed, when the zero-order rate was approximately doubled.

Iron is more important as a catalyst for the synthesis of liquid fuels than cobalt or nickel because of lower cost and added flexibility of the syntheses. The iron carbides are almost always formed in the synthesis. The mode of transition from one carbide to the other in the reaction

$$\underset{\text{Hägg carbide}}{3Fe_2C} \longrightarrow \underset{\text{Cementite}}{2Fe_3C} + \underset{\text{Free carbon}}{C}$$

was studied on Hägg carbide, prepared from a precipitated iron catalyst, by successive heat treatments at 525° C. for increasing periods of time.[95] After each treatment the sample was cooled rapidly to 300° C. to suppress further reaction, then cooled very slowly in the range of the magnetic transformations while magnetic measurements were made at frequent intervals. The reaction was found to be a discontinuous precipitation. Hägg carbide and cementite evidently have narrow ranges of composition and do not form intermediate metastable solid solutions. The reaction

$$\underset{\text{Hägg carbide}}{Fe_2C} + Fe \longrightarrow \underset{\text{Cementite}}{Fe_3C}$$

produces cementite in better yields than previously known methods of preparation, and the cementite is pure, unsintered, and suitable for use in the Fischer-Tropsch synthesis.[96] This reaction requires substantially lower temperatures than any other known series of reactions producing cementite and is relatively simple to carry out. When a mixture of Hägg carbide and iron was heated in vacuum to about 450° C. and held at that temperature, the reaction went almost to completion in 1.5 hours.

Analysis of Iron Catalysts

A convenient method for the analysis of used iron catalysts was developed[97] in which the use of thermomagnetic measurements, made with the magnetic balance, in connection with X-ray diffraction and chemical analyses gave data from which the relative amounts of the ferromagnetic phases could be calculated. About 30 percent of the catalyst could not be accounted magnetically. This portion of unknown composition may result from magnetic unsaturation, and some may be finely divided or in an otherwise magnetically hard state. This latter may play a part in the synthesis.

The relative amounts of iron and its compounds were determined as a function of catalyst life by the magnetic method.[98] The catalyst was periodically removed and

95/ Cohn, E. M., and Hofer, J. J. E., Mode of Transition from Hägg Iron Carbide to Cementite: Jour. Am. Chem. Soc., vol. 72, October 1950, pp. 4662-4664.

96/ Cohn, E. M., and Hofer, L. J. E., Preparation of Iron Carbides: U. S. Patent 2,535,042, December 1950.

97/ Hofer, L. J. E. and Cohn, E. M., Thermomagnetic Determination of Hägg Carbide in Used Iron Fischer-Tropsch Catalysts: Anal. Chem., vol. 22, July 1950, pp. 907-910.

98/ Anderson, R. B., Hofer, L. J. E., Cohn, E. M., and Seligman, B., Studies in the Fischer-Tropsch Synthesis. IX. Phase Changes of Iron Catalysts in the Synthesis: Jour. Am. Chem. Soc., vol. 73, March 1951, pp. 944-946.

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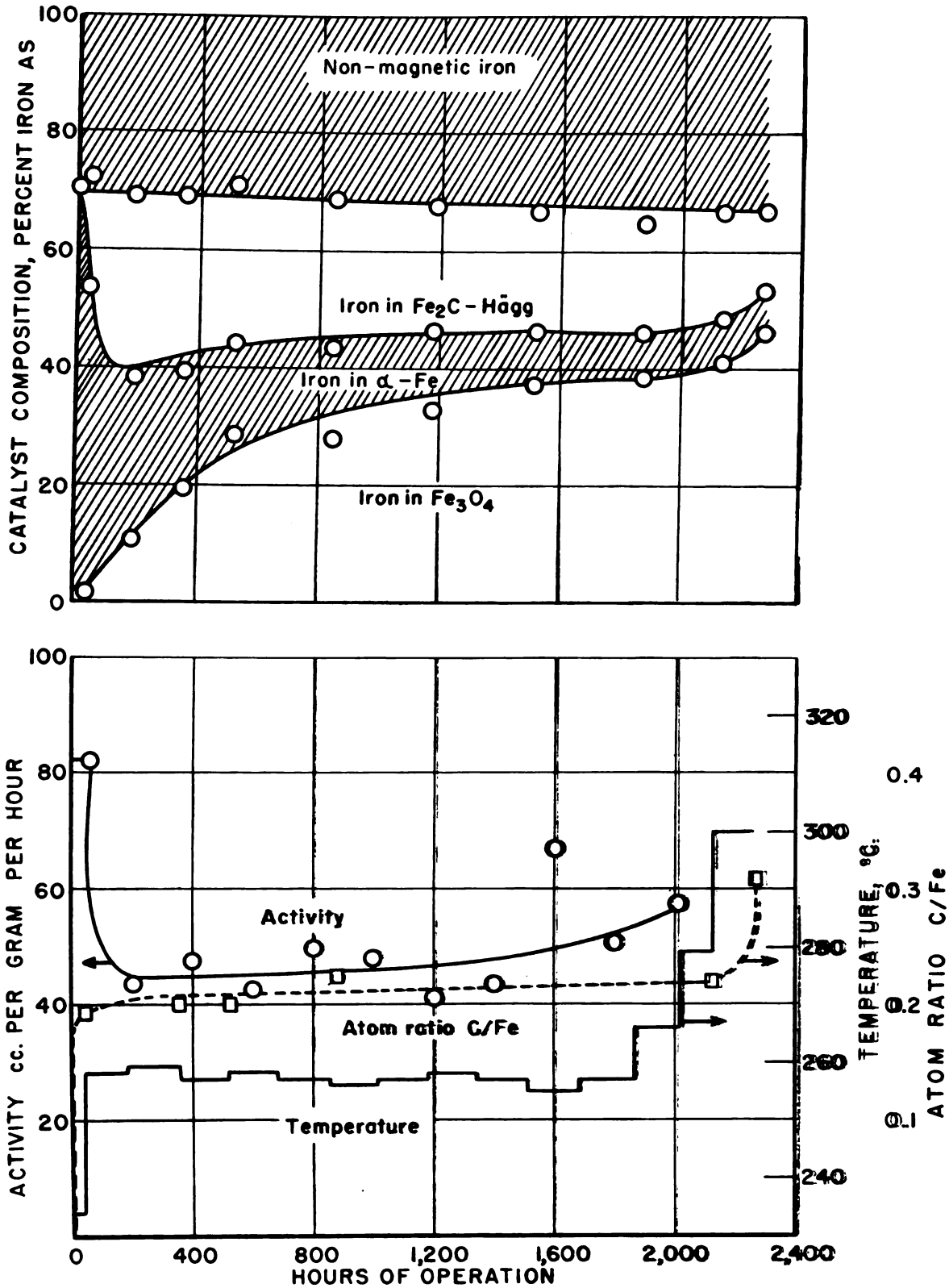

Figure 20. - The variation of catalyst composition, activity, and temperature with time: Fe_3O_4-MgO-K_2O catalyst at 7.8 atmospheres of $1H_2$: 1CO gas.

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sampled, care being taken to avoid its exposure to the atmosphere. The original reduced catalyst contained only metallic and "nonmagnetic" iron. As shown in figure 20, the amount of the latter remained essentially constant at about 30 percent throughout the synthesis. While the amount of metallic iron decreased continuously, Hägg carbide formation reached a maximum in about 8 days, after which it was oxidized at a considerably slower rate than iron.

Nitrided Iron Catalysts

Treating iron with ammonia to convert it to iron nitride resulted in catalysts of enhanced activity and longer life that produced large amounts of oxygenated products.99/ Depending on the ammonia treatment, various iron nitrides are formed. These are interstitial compounds similar to, and in some cases isomorphous with, the carbides of iron.

Nitrided fused catalysts operated at a considerably lower temperature than the same reduced catalysts. In a life test one nitrided catalyst operated for 190 days at 7.8 atmospheres; at the end of that time the temperature of operation was below that of a reduced catalyst, and the catalyst was still in good condition. Tests with initially reduced catalysts were usually ended in much shorter periods because of oxidation or plugging of the catalyst bed by carbon or wax deposits. The operation of nitrided catalysts at 21.4 atmospheres pressure was characterized by nearly constant temperature of operation, but the increase in activity over that of the reduced catalyst was not as marked as for the operation at 7.8 atmospheres. Greater yields of gaseous hydrocarbons and oxygenated compounds, including alcohols, were obtained with nitrided catalysts than with reduced catalysts. The difference in selectivity must be related to a specific change of the catalyst surface. The tendency of nitrided catalysts to resist oxidation and free carbon deposition may contribute to the longer life of these catalysts.

Surface Area and Pore Volume Studies

As part of the research on the changes that occur in iron catalysts during pretreatment or during synthesis, the changes in surface areas and pore volumes of a precipitated and of a fused iron catalyst have been investigated.1/ The two catalysts differed widely in their original form, the precipitated catalyst, being a ferric oxide gel, had a high surface area before reduction, while the unreduced fused-iron oxide catalyst had a small surface area. After reduction in hydrogen, the two catalysts had similar surface areas (6-10 $m.^2/gm.$) and pore volumes (.09-.16 cc./gm.).

When the precipitated catalyst was sintered in nitrogen at increasing temperatures up to 550° C., the pore volume reached a maximum of 0.151 cc./gm. at 300° C. and then decreased, while the surface area decreased progressively. The average pore diameter increased with increasing sintering temperatures. Reduction of the fused catalyst resulted in an increase in surface area because of the removal of oxygen, although the external volume did not change. Up to 90 percent reduction, the pore volume and surface area increased linearly with extent of reduction.

99/ Anderson, R. B., Shultz, J. F., Seligman, B., Hall, W. K., and Storch, H. H., Studies of the Fischer-Tropsch Synthesis. VII. Nitrides of Iron as Catalysts: Jour. Am. Chem. Soc., vol. 72, August 1950, pp. 3502-3508.

1/ Hall, W. K., Tarn, W. H., and Anderson, R. B., Studies of the Fischer-Tropsch Synthesis. VIII. Surface Area and Pore Volume Studies of Iron Catalysts: Jour. Am. Chem. Soc., vol. 72, December 1950, pp. 5436-5443.

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When the reduction temperature was increased to 650° C., the surface area was reduced to about one-sixth of the area of the same catalyst reduced at 450° C., and the average pore diameter increased eightfold. At 650° C. the promoters still retarded but could not prevent sintering of the newly developed surface, as they had at lower temperatures. The reduced fused catalysts had higher surface areas than reduced pure magnetite. For preparations of this type, variation of the reduction temperature affords a method for producing catalysts with predetermined pore diameters. This effect may be of considerable practical importance, as the activity of the catalyst is related to the pore diameter.

Pilot-Plant Developments

Oil-Circulation Processes

Bureau of Mines developments in the oil-circulation Fischer-Tropsch process have been to improve existing processes by eliminating the features that cause catalyst disintegration and cementing.2/ The moving catalyst bed, developed to eliminate the cementing problem in fixed-catalyst beds, operated for several months without an increase in pressure drop across the bed and without cementing of the catalyst. The apparatus and controls are shown in figure 21. Decrease in catalyst activity and carry-over of catalyst into the oil-circulation lines were negligible. Other advantages of the moving bed over the fixed-bed process were found to be: Greater catalyst activity per unit mass due to the use of finer particles (that is, due to larger geometric surface area per weight of catalyst); lower operating temperatures; and easier charging and withdrawal of catalyst. A 91 percent over-all synthesis-gas conversion is obtainable, using 1.3:1 hydrogen to carbon monoxide synthesis gas, by operating a two-stage process with 70 percent conversion in each stage and with a recycle ratio of tail gas to fresh synthesis gas of 1:1. A rapid decline in catalyst activity occurred when 25 percent carbon dioxide was added to the feed gas to simulate operation of a second stage without removal of carbon dioxide. No attempt was made to determine the maximum catalyst life, but it was noted that one batch of catalyst still possessed considerable activity after about 2,700 hours of synthesis.

As compared with fixed-bed runs, the moving-bed experiments yielded smaller amounts of gaseous hydrocarbons and oxygenated products. The gas recycle ratio (volume of tail gas to fresh gas) was varied to study the effect on the usage ratio (volume of hydrogen reacted per volume of carbon monoxide) of synthesis gas and on the production of hydrocarbon gas. As the recycle ratio increased, the gas production decreased, and the hydrogen to carbon monoxide usage ratio rose. Both of these trends are desirable for efficient conversion of synthesis gas.

Feed gas with a hydrogen to carbon monoxide ratio of 1:1 can be used. This is more economically produced from coal than hydrogen-rich gas and may offer the best possibility for achieving more economical synthesis gas usage, as the cost of purified synthesis gas, produced from coal, has been estimated to be about 60-70 percent of the total cost of the synthesis.

Slurry-Phase Operation

In the slurry-phase method for the Fischer-Tropsch synthesis, finely divided catalyst is suspended in oil, and the reaction gas is bubbled through the oil at a

2/ Crowell, J. H., Benson, H. E., Field, J. H., and Storch, H. H., Fischer-Tropsch Oil Circulation Processes: Ind. Eng. Chem., vol. 42, November 1950, pp. 2376-2384.

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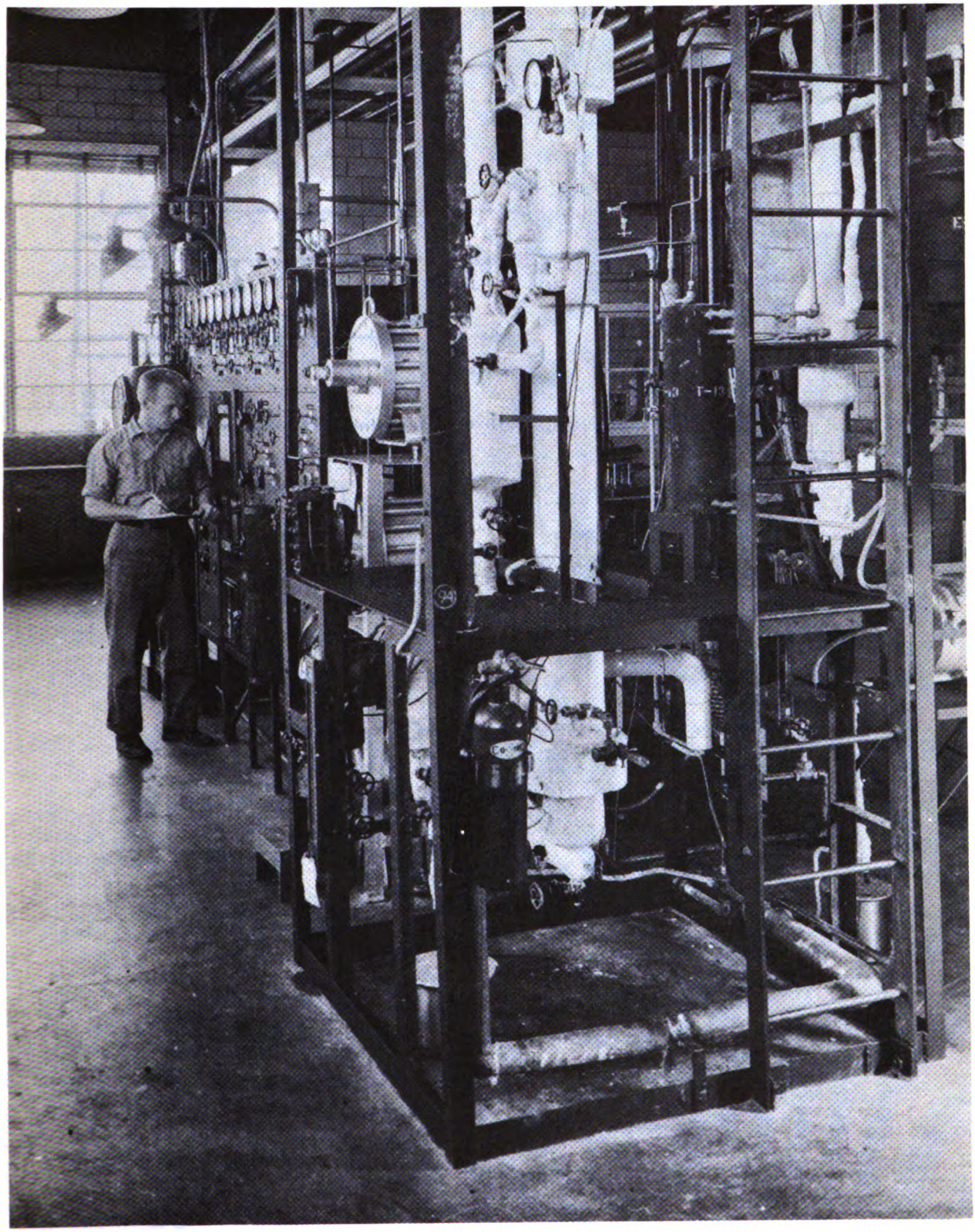

Figure 21. - Large catalyst-oil slurry unit.

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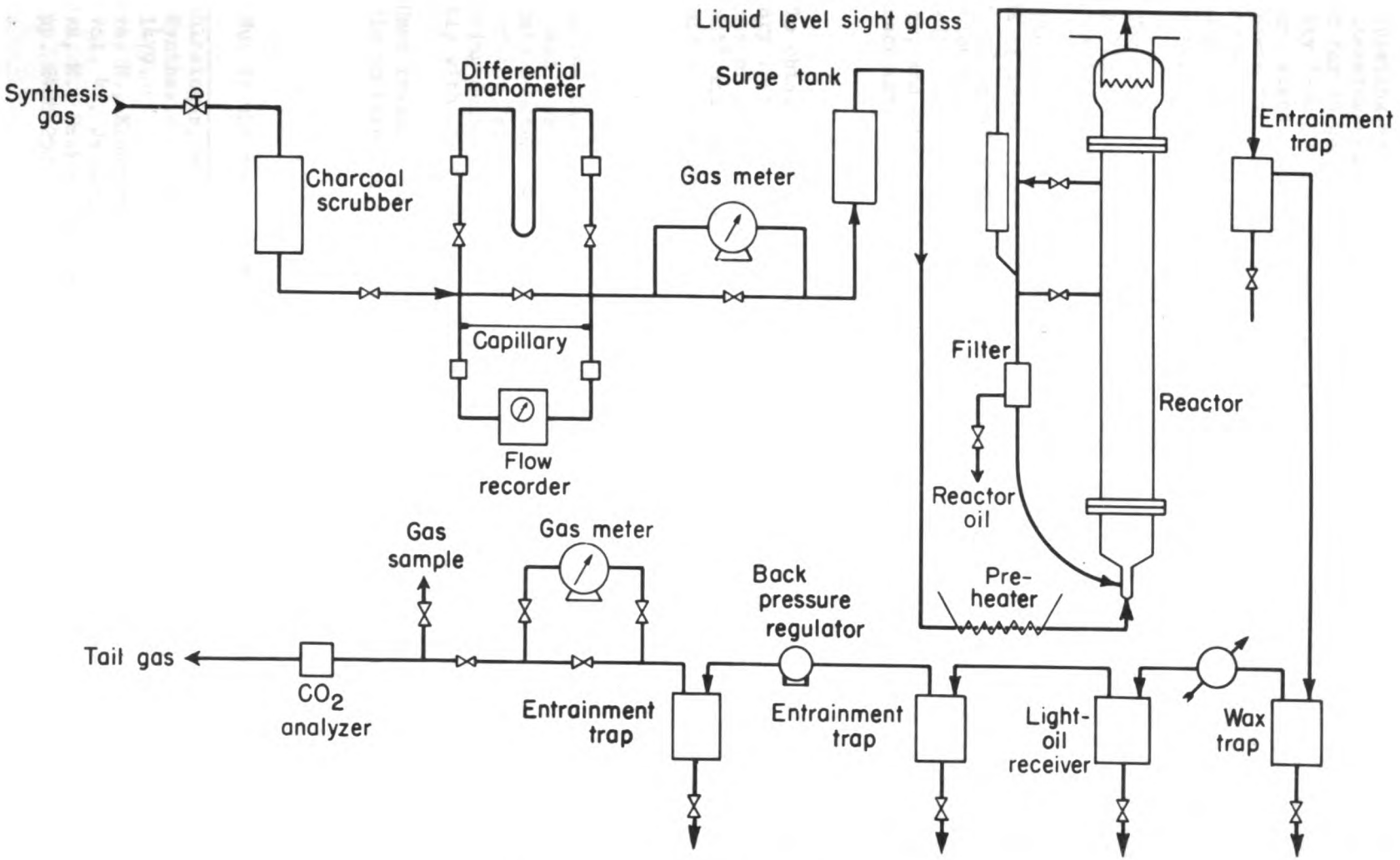

Figure 22. - Flow diagram of oil slurry process.

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rate rapid enough to keep the catalyst in suspension. Orienting pilot-plant tests in a circulating system, as diagramatically shown in figure 22, were made with a copper- and potassium-promoted precipitated iron catalyst.3/ During the operation, which lasted for 75 weeks, the pressure was varied from 100 to 250 p.s.i.g., the space velocity from 130 to 300, and the temperature from 242° to 276° C. The activity declined steadily during the test, requiring increases in temperature or pressure to compensate for the deactivation. Catalyst settling was a contributing mechanical cause for decreased activity, as was found by the determination of the slurry densities at different bed heights and time intervals. A slow, continuous increase in the carbon content of the catalyst was observed.

The production of light hydrocarbons increased with temperature. Pressure had a negligible influence on the extent of methane formation but augmented the carbon monoxide conversion, especially below 200 p.s.i.g.

The over-all conversion of synthesis gas was 55 percent, resulting in average yields of 12 gm. (per cubic meter of inlet gas) of gaseous hydrocarbons ($C_1 + C_2$), 90 gm. of C_3 and higher hydrocarbons, and 12 gm. of water. The average wax content (fraction boiling above 450° C.) of the slurry was more than 50 percent. Of the reacted carbon monoxide, 5 percent was converted to $C_1 + C_2$, 43 percent to C_3 and heavier, and 42 percent to carbon dioxide. About 10 percent of the carbon monoxide was unaccounted, partly because no detailed analysis of oxygenated compounds was made.

Fixed- and Fluidized-Bed Studies

To obtain engineering data on the flow of gases through beds of catalysts, preliminary investigation of the physical phenomena of elutriation was made,4/ using mixtures of particles of different diameters, the coarser particles serving as "solvent" and the fines as "solute." Within certain ranges of concentration of fines in fluidized mixtures of sand and of fused iron catalyst,

$$c = c_o e^{-2.303\ kt},$$

where c was the concentration of fines at time t, c_o was the initial concentration of fines, and k was a quasi-first-order specific reaction rate constant. This elutriation rate constant was rather insensitive to changes in the composition and range of sizes of the "solvent," but it increased rapidly with increasing average diameter and decreasing density of the "solute." Within the range of conditions tested, it varied directly with the fourth power of the linear rate of gas flow.

Heat transfer in packed tubes was studied with equipment having high ratios of particle to tube diameter5/ and the following empirical correlation was found:

$$\frac{Nu'}{Re^{0.75}} = 0.125,$$

where Nu' is the modified Nusselt number, and Re is the modified Reynolds number.

3/ Schlesinger, M. D., Crowell, J. H., Leva, M., and Storch, H. H., Fischer-Tropsch Synthesis in the Slurry Phase: Ind. Eng. Chem., vol. 43, June 1951, pp. 1474-1479.

4/ Leva, M., Elutriation of Fines from Fluidized Systems: Chem. Eng. Progress, vol. 47, January 1951, pp. 39-45.

5/ Leva, M., Packed Tube Heat Transfer: Ind. Eng. Chem., vol. 42, December 1950, pp. 2498-2501.

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Within experimental limits, the tube diameter had no effect upon the heat-transfer coefficients, which were surprisingly high as compared with those predicted by a correlation based on small ratios of particle size to tube diameter. Increasing the particle size caused a significant reduction in pressure drop, and the heat-transfer to pressure-drop relationship agreed with available data. A qualitative evaluation of the wall effect showed that fluid channeling between catalyst bed and wall (and thus catalyst by-passing) became increasingly severe as the ratio of particle diameter to tube diameter increased. Therefore, the use of high ratios is undesirable for contact catalysis where contact between solid and gas and effective utilization of the latter are of prime importance.

Production of Alcohols

In the reaction, wherein an alcohol is treated with synthesis gas (carbon monoxide and hydrogen) in the presence of a cobalt catalyst to produce a primary alcohol containing one more carbon atoms than the starting material, the order of reactivity is usually tertiary > secondary > primary. However, methyl alcohol is anomalous in that it reacts with synthesis gas more rapidly than do secondary alcohols.6/ The chief product of this reaction is ethyl alcohol, which can be homologated further only with difficulty. This method of making ethanol is not only of technical interest but is also of theoretical value, as the reaction cannot proceed via an olefin intermediate. Furthermore, the product distribution from this reaction was similar to that of the oxygenated fraction of Fischer-Tropsch synthesis products.

Higher alcohols can be obtained from aldehydes and ketones7/8/9/ by the same type of reaction. The catalysis appears to be homogeneous and to go by way of cobalt hydrocarbonyl or dicobalt octacarbonyl, as it is not affected adversely by sulfur compounds, which inhibit hydrogenation on a solid catalyst. In systems containing conjugated double bonds, both hydrogenation and hydroformylation occur. The former becomes the dominant reaction as the degree of double-bond character of an olefinic linkage diminishes because of conjugation. There must be adequate concentration of carbon monoxide in the reacting mixture to form cobalt carbonyl. If the partial pressure of carbon monoxide is below this threshold value, the cobalt metal may be poisoned for any catalytic activity or - in the absence of carbon monoxide - heterogeneous hydrogenation on metallic cobalt may proceed.

Oxygen Exchange Between Nitrates and Water

In a cooperative project with the Explosives and Physical Sciences Division, Region VIII, the mass spectrometer was used for the analysis of the oxygen exchange between nitrates and water.10/ Water enriched with O^{18} exchanged its tagged oxygen with a 40-percent solution of nitric acid at 30° C., but did not undergo exchange with cellulose nitrate 1N sulfuric acid solution at 23° and 100° C., as determined by analysis of the water.

6/ Wender, I., Friedel, R. A., and Orchin, M., Ethanol from Methanol: Science, vol. 113, February 1951, pp. 206-207.

7/ Wender, I., Levine, R., and Orchin, M., Chemistry of the Oxo and Related Reactions. II. Hydrogenation: Jour. Am. Chem. Soc., vol. 72, October 1950, pp. 4375-4378.

8/ Wender, I., Orchin, M., and Storch, H. H., Mechanism of the Oxo and Related Reactions. III. Evidence for Homogeneous Hydrogenation: Jour. Am. Chem. Soc., vol. 72, October 1950, p. 4842.

9/ Wender, Irving, Greenfield, Harold, and Orchin, Milton, Chemistry of the Oxo and Related Reactions. IV. Reductions in the Aromatic Series: Jour. Am. Chem. Soc., vol. 73, No. 6, June 1951, pp. 2656-2658.

10/ Klein, R. and Friedel, R. A., Oxygen Exchange Between Nitrates and Water: Jour. Am. Chem. Soc., vol. 72, August 1950, pp. 3810-3811.

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SYNTHETIC LIQUID FUELS BY COAL HYDROGENATION: RESEARCH AND DEVELOPMENT

Batch Autoclave Studies

Mechanism of Coal Hydrogenation

A study[11] in small autoclaves of the hydrogenation and pyrolysis of anthraxylon from bituminous coal has been made as part of the program to elucidate the mechanism of coal hydrogenation. Table 14 shows the product distribution for hydrogenation and pyrolysis of anthraxylon, where the hydrogenation was carried out in a hydrogen atmosphere and the pyrolysis in a nitrogen atmosphere. In the absence of catalyst, the conversion of anthraxylon to asphalt, gas, and oil was very low. The carbon dioxide formation was small, yet greater than in the presence of catalyst (1 percent tin plus 0.5 percent ammonium chloride). Elimination of oxygen as water and extent of liquefaction were increased by the presence of a catalyst. However, the amount of liquefaction alone does not determine the degree of oxygen elimination. Apparently, liquefaction is always accompanied by the formation of a large amount of water, but the converse is not true.

TABLE 14. - Product distributions for hydrogenation and pyrolysis of anthraxylon[1]

Run No.	Temperature, °C.	Catalyst	Moisture and ash-free coal, percent: Organic benzene-insolubles	Asphalt	Oil	Hydrocarbon gases	CO_2	H_2S	H_2O	H_2 absorbed
Hydrogenation										
1,180.....	400	-	89.75	2.57	2.98	2.37	0.82	0.10	1.21	0.03
1,320.....	400	2/	24.89	61.01	8.13	4.63	.51	.10	3.82	2.54
1,284.....	450	-	73.16	5.66	7.82	8.23	1.13	-	3.21	.66
1,219.....	450	2/	13.96	40.84	28.09	12.96	.20	.10	5.72	3.37
Pyrolysis										
1,193.....	400	-	92.83	-	2.88	2.12	0.72	0.10	1.29	-0.32
1,191.....	400	2/	91.46	-	2.88	2.16	.62	-	.71	- .73
1,206.....	450	-	88.33	-	4.94	3.12	1.13	-	1.59	- .56
1,192.....	450	2/	86.93	-	4.73	3.60	1.13	-	2.08	- .69

1/ 100 gm. Bruceton anthraxylon, 1 hr., 1,000 lb./in.^{2} initial hydrogen or nitrogen pressure.

2/ 1 percent tin plus 0.5 percent ammonium chloride.

The first step in the over-all hydrogenolysis of coal may be a splitting reaction resulting in reactive fragments. These fragments can either be stabilized by adding hydrogen to form benzene-soluble products or they can be polymerized, without hydrogenation, to form benzene-insoluble products. The results of an uncatalyzed run tend to support this hypothesis, as low hydrogen consumption, low production of water and hydrocarbon gas, and high production of benzene-insolubles were observed. The small extent of liquefaction obtained with anthraxylon under these conditions may be interpreted as resulting from a slow rate of hydrogenation of fragments compared to their rate of polymerization. If the theory is correct, the production of much of the water and of hydrocarbon gas during coal hydrogenation must be associated with some process other than the first step in which reactive fragments are formed.

11/ Pelipetz, M. G., Weller, S., and Clark, E. L., Hydrogenation and Pyrolysis of Anthraxylon from Bituminous Coal: Fuel, vol. 29, September 1950, pp. 208-211.

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The above hypothesis was further substantiated by studying the pyrolysis of coal under the same conditions and in the same equipment as used for hydrogenation, but with a nitrogen atmosphere instead of hydrogen. As shown in table 14, only small amounts of gas, water, and oil were formed, and virtually no asphalt was produced. The course of the pyrolysis was essentially unaffected by the presence of a catalyst, and the effect of temperature was small. As primary splitting appears to take place in the same temperature range during pyrolysis, these data support the assumption that much of the water and gas production results from secondary reactions. The former probably is the result of the reaction of oxygen-containing groups with gaseous hydrogen.

Catalysts for Coal Hydrogenation

The activity of a number of catalysts was much greater when they were impregnated on the coal from aqueous solution than when they were added as powders to powdered coal.12/13/ Ball-milling of coal and catalyst together was of intermediate effectiveness. Thus, if the catalyst is evenly distributed throughout the coal, the efficiency of many hydrogenation catalysts can be significantly improved, and cheaper and more readily available catalysts utilized. The unique position of tin compounds as hydrogenation catalysts no longer exists, and the explanation of their high activity in powdered form must be sought in an as yet unknown mechanism, which permits good distribution of tin compounds, regardless of the form in which they are added.

Pilot-Plant Construction: Vapor-Phase Hydrogenation Unit

To provide equipment for the hydrogenation of oils over a fixed catalyst bed, a small plant suitable for operation at 500° C. and 10,000 p.s.i.g. has been designed.14/ A simplified flow diagram of the unit is shown in figure 23. Special connections and valves were designed for the unit. All high-pressure equipment, except compressor and pump, was enclosed in an open-topped stall with concrete walls. The performance of the unit was tested, using an aromatic oil (boiling below 325° C.) as the raw material. This was obtained by distillation of a blend of light oils originating from coal that had been hydrogenated in the liquid-phase. Product analyses and material balances indicated that a catalyst consisting of small amounts of chromium, molybdenum, and zinc oxides deposited on fuller's earth activated with hydrogen fluoride was suitable for the hydrogenation, in the course of which the oxygen sulfur, and nitrogen contents were reduced. The gasoline produced, which was approximately 50 percent of the starting material, retained the original aromatic structure and possessed good antiknock characteristics. The results indicated the suitability of the plant for performing high-pressure catalytic reactions.

Separation and Identification of Products of Coal Hydrogenation

X-Ray Diffraction Analysis

Solid crystalline, aromatic hydrocarbons can be identified by X-ray diffraction powder patterns, in which closely related compounds, even isomers, can be distinguished,

12/ Weller, S. and Pelipetz, M. G., Coal-Hydrogenation Catalysts. Studies of Catalyst Distribution: Ind. Eng. Chem., vol. 43, May 1951, pp. 1243-1246.

13/ Weller, S. and Pelipetz, M. G., Catalysis in Liquid-Phase Coal-Hydrogenation: Proc. Third World Petrol. Cong., The Hague, Netherlands, sec. IV, subsec. I, preprint 7, June 3, 1951, 6 pp.

14/ Wolfson, M. L., Pelipetz, M. G., Damick, A. D., and Clark, E. L., Vapor-Phase Hydrogenation of Light and Heavy Oils: Ind. Eng. Chem. vol. 43, February 1951, pp. 536-540.

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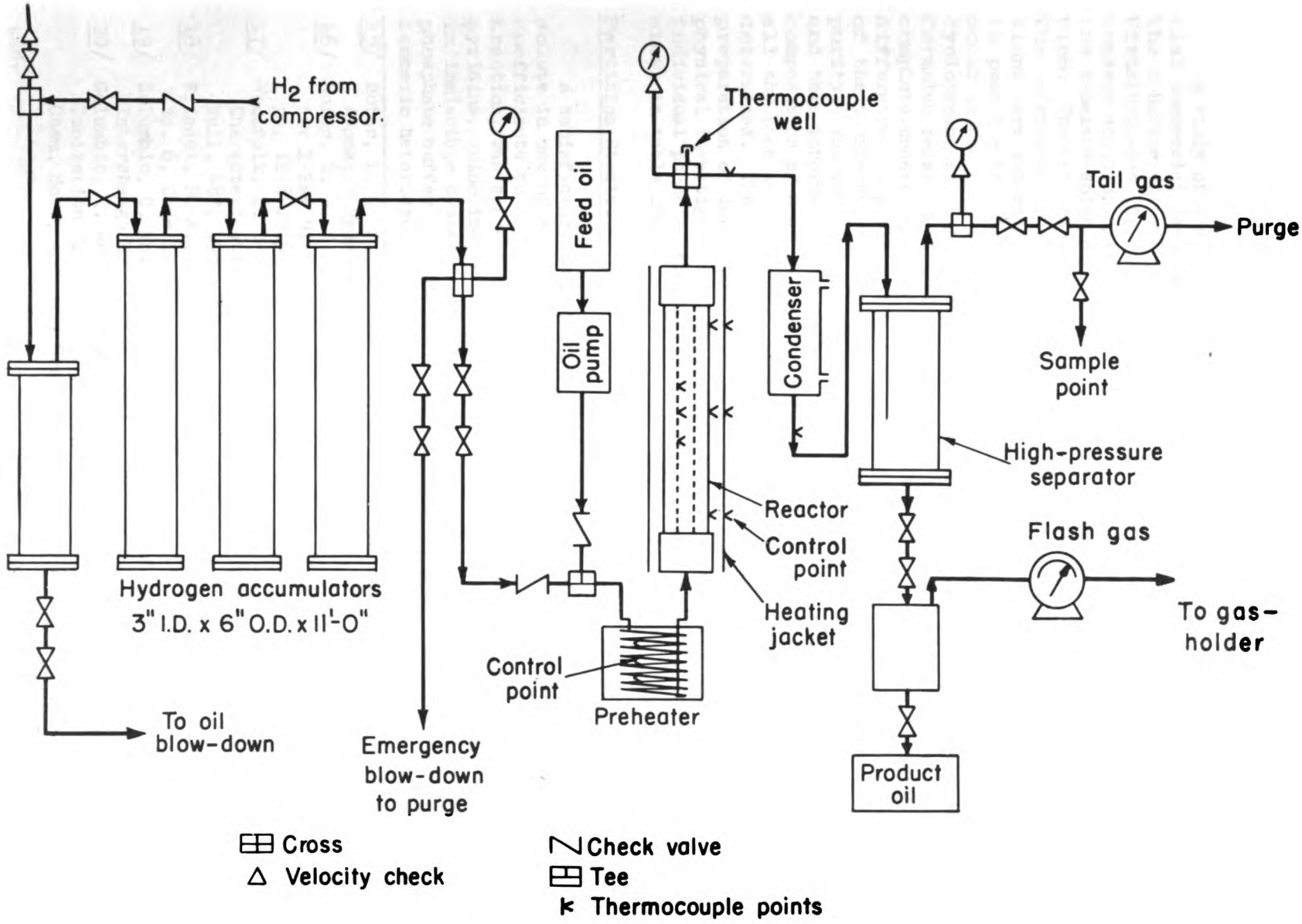

Figure 23. - Flow diagram of vapor-phase hydrogenation unit.

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while impurities adequate to lower the melting point by as much as 15° C. do not appreciably modify the patterns.[15] A sample-preparation method was developed,[16] and the patterns of 59 hydrocarbons were obtained. The method is applicable to microsamples and is nondestructive.

Studies of Phenols in Coal-Hydrogenation Oils

A study of the tar acids in coal-hydrogenation oils[17] deals with their potential commercial importance, occurrence, and separation. The tar-acid fraction of the n-hexane-soluble portion of the liquid product obtained by the hydrogenation of Pittsburgh-bed (Bruceton) coal was isolated by methanolic alkali extraction of the benzene solution of the oil. The yield of tar acids was 9.4 percent by weight of the n-hexane-soluble oil, or 5.2 percent of the total oil produced in the hydrogenation. The tar acids were subjected to distillation, and 136 fractions were collected. The refractive index of each fraction and the densities of the higher-boiling fractions were determined. Careful interpretation of the data on physical constants makes it possible to select those fractions that are rich in any desired phenol known to occur in the oil. A study of the distribution (partition) of various phenols between cyclohexane and a buffered aqueous phase led not only to the development of rules and formulas relating partition coefficient to ionization constant but also to a more complete understanding of the factors governing the extractability of phenols. The differences in partition coefficients of isomeric phenols permitted the application of the countercurrent distribution method to their separation and determination of purity. The infrared spectra of many phenols of known constitution were determined,[18] and this information was applied to the qualitative and quantitative estimation of the composition of most of the tar-acid fractions. The ultraviolet absorption spectra of all the phenols known to occur in the oil obtained from the hydrogenation of coal were determined. The more classical methods of organic chemistry, such as crystallization, preparation of derivatives, and degradative studies, also were used. Chemical and physical investigation of the various fractions resulted in the identification of 16 individual phenols, as well as inconclusive evidence of the presence of 2 others, as shown in table 15.

Partition Studies

A satisfactory method that involves a means of increasing the concentration of solute in one of the solvents was developed[19] for measuring large or small partition coefficients to obtain measurable amounts of solute. The technique, interchange extraction, was subjected to precise mathematical analysis. The distributions of pyridine, quinoline, and certain of their methyl-substituted derivatives were studied in immiscible systems composed of cyclohexane or chloroform and of water or citrate-phosphate buffer.[20] The data thus obtained will be useful for the separation of isomeric heterocyclic bases by countercurrent distribution.

15/ Hofer, L. J E., and Peebles, W. C., X-Ray Diffraction Patterns of Solid Aromatic Hydrocarbons: Anal. Chem., vol. 23, May 1951, pp. 690-695.

16/ Hofer, L. J. E., Peebles, W. C., and Guest, P. G., Preparing Extruded Samples for X-Ray Diffraction Analysis: Anal. Chem. vol. 22, September 1950, pp. 1218-1219.

17/ Woolfolk, E. O , Golumbic, C., Friedel, R. A., Orchin, Milton, and Storch, H. H., Characterization of Tar Acids from Coal-Hydrogenation Oils: Bureau of Mines Bull. 487, 1950, 56 pp.

18/ Friedel, R. A., Infrared Spectra of Phenols: Jour. Am. Chem. Soc., vol. 73, No. 6, June 1951, pp. 2881-2884.

19/ Golumbic, C. and Weller, S., Measurement of Large Partition Coefficients by Interchange Extraction: Anal. Chem., vol. 22, November 1950, pp. 1418-1419.

20/ Golumbic, C. and Orchin, M., Partition Studies. V. Partition Coefficients and Ionization Constants of Methyl-Substituted Pyridines Quinolines: Jour. Am. Chem. Soc., vol. 72, September 1950, pp. 4145-4147.

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TABLE 15. - Tar acids found in the oil from hydrogenation of Bruceton coal

Compound	Hexane-soluble oil, percent	Total oil,	Pounds per ton of total oil	Pounds per ton of coal[1]
Phenol	0.24	0.13	2.6	2.0
o-Cresol	.37	.20	4.0	3.1
m-Cresol	.55	.30	6.0	4.7
p-Cresol	.25	.14	2.8	2.2
2,4-Xylenol	.23	.13	2.6	2.0
2,5-Xylenol	.14	.08	1.6	1.2
3,5-Xylenol	.25	.14	2.8	2.2
3.4-Xylenol	.05	.03	.6	.5
2,3-Xylenol[2]	-	-	-	-
o-Ethylphenol	.03	.02	.4	.3
m-Ethylphenol	.42	.23	4.6	3.6
p-Ethylphenol	.09	.05	1.0	.8
Mesitol[2]	-	-	-	-
3-Methyl-5-ethylphenol	.27	.15	3.0	2.3
4-Indanol	.19	.10	2.0	1.6
5-Indanol	.13	.07	1.4	1.1
o-Phenylphenol[3]	-	-	-	-
p-Phenylphenol[3]	-	-	-	-
Total	3.21	1.77	35.40	27.60

1/ Dry, ash-free coal.
2/ Presence indicated by chemical methods, but presence could not be confirmed spectroscopically.
3/ Definitely shown to be present, but quantity not estimated.

The phenolic constituents of asphalt obtained by coal hydrogenation also were examined by the countercurrent-distribution method.[21] As defined here, asphalt is the benzene-soluble, n-hexane-insoluble portion of the coal-hydrogenation product. Two groups of phenols were distinguished: One group, extracted by aqueous alkali, comprised alkyl phenols and phenyl phenols; the other group, soluble only in methanolic alkali, was probably a mixture of polynuclear phenols. The oxygen content of the latter group amounted to about one-third of the total oxygen in the asphalt, most of which was present in hydroxyl groups. Only about 30 percent of the oxygen in the nonextracted fraction could be ascribed definitely to hydroxyl groups.

Organic Syntheses

The synthesis of organic compounds[22][23][24] was continued as part of the program to study the structure of coal and to identify the products of its hydrogenation.

21/ Husack, R. and Golumbic, C., The Characterization of the Phenolic Fraction of Coal-Hydrogenation Asphalt: Jour. Am. Chem. Soc., vol. 73, April 1951, pp. 1567-1571.
22/ Orchin, M., Reggel, L., and Friedel, R. A., The Anomalous Reaction of 1-Tetralone with o-Tolylmagnesium Bromide: Jour. Am. Chem. Soc., vol. 73, April 1951, pp. 1449-1454.
23/ Orchin, M., and Reggel, L., Aromatic Cyclodehydrogenation: X. Studies on Ring Oxygen Compounds. Dinaphtho 1, 2, 1', 2' -furan: Jour. Am. Chem. Soc., vol. 73, April 1951, pp. 1877-1878.
24/ Orchin, Milton and Reggel, Leslie, A Synthesis of Fluoranthene by Cyclohydrogenation: Jour. Am. Chem. Soc., vol. 73, No. 6 June 1951, pp. 2955, 2956.

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Methods for the preparation of two isomers of benzfluoranthene were developed in another synthesis study.[25]

The distillation laboratory at Bruceton is used for separating and identifying products obtained by the synthetic liquid fuel processes. The laboratory was designed for flexibility and versatility of operation[26] because a large variety of organic compounds must be handled. These were achieved, in part, by standardization of basic equipment, permitting interchange of parts and maximum service.

The effect of pressure and of the type of packing on the efficiency of packed distillation columns was studied by measuring fractionating efficiencies, throughputs, and holdups at various reflux rates.[27] Pressures from 20 to 760 mm.-Hg, with Berl saddles, single- and triple-turn helices, glass spheres, and a commercial Heli-grid column packing, were used. In general, efficiencies decreased with decreasing pressure below about 200 mm.-Hg. The fixed-Heli-grid packing was superior to all of the loose packings that were tested; of the latter, glass spheres gave the best results. Columns filled with loose packings were most efficient when operated near maximum throughput capacity; their highest separating power appears to be near 200 mm.-Hg.

Permeation of Gases Through Membranes

The cost of gas is an important factor in the production of synthetic liquid fuel. Oxygen is needed for the gasification of coal, and hydrogen for use in the synthesis gas mixture or for use in the hydrogenation of coal. One of the most promising methods for the production of these gases, at a cost competitive with commercial methods, is the enrichment of hydrogen or oxygen in gas mixtures by fractional or differential permeation through nonporous membranes. Of 15 nonporous films tested in a laboratory-scale apparatus, natural rubber was outstanding in showing the highest permeability toward the gases tested. Its selectivity as between gases, however, was not as great as that of several other films, and it was subject to oxidation, with consequent change in properties. Ethyl cellulose best satisfies the requirements of high permeability and high selectivity. The permeability of the membranes for each gas tested (nitrogen, oxygen, hydrogen, helium) increased with increasing temperature. The temperature dependence (and therefore the activation energy for permeation) is about the same for oxygen, helium, and hydrogen, but is greater for nitrogen. For the enrichment of gas mixtures, a compromise must be made between the use of higher temperatures to obtain higher permeability and lower temperatures to obtain greater selectivity. The use of a fractional permeation process may be of practical importance[28][29][30] to effect such separations as oxygen from air, helium from natural gas, and hydrogen from hydrogenation tail gas. As the permeable films are not perfectly selective, it is

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25/ Orchin, M., and Reggel, L., Synthesis of Benz [j] Fluoranthene and Benz [k] Fluoranthene: Jour. Am. Chem. Soc., vol. 73, January 1951, pp. 436-442.

26/ Feldman, J., Pantazoplos, P., Pantazoplos, G., and Orchin, M., Design, Construction, and Operation of a Distillation Laboratory for the Synthetic Liquid Fuels Program: Bureau of Mines Rept. of Investigations 4764, 1951, 4 pp.

27/ Myles, M., Feldman, J., Wender, I., and Orchin, M., Fractionating Efficiency of Various Packings: Ind. Eng. Chem., vol. 43, June 1951, pp. 1452-1456.

28/ Weller, S. and Steiner, W. A., Engineering Aspects of Separation of Gases. Fractional Permeation through Membranes: Chem. Eng. Prog., vol. 46, November 1950, pp. 585-590.

29/ Weller, S. and Steiner, W. A., Separation of Oxygen from Gas Mixtures Containing the Same: U. S. Patent 2,540,151, Feb. 6, 1951.

30/ Weller, S., Recovery of Light Elemental Gases: U. S. Patent 2,540,152, Feb. 6, 1951.

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necessary, in general, to employ a multistage recycle system to obtain gases of high purity. A simplified flow diagram for a three-stage permeation process is shown in figure 24.

For appropriate gas mixtures, the power requirements of a permeation process are in the same order of magnitude as for low-temperature separation. Relatively large plants will be required because of low film permeabilities. The total film area (that is, plant size) needed is approximately inversely proportional to the pressure used on the high-pressure side. An increase in pressure therefore will decrease the film area that is needed. According to available data, production of pure oxygen by a permeation process is not competitive with the Linde process because of high power requirements and large-plant size. The production of slightly enriched air, helium from natural gas, and the enrichment of hydrogen-containing gases seem more promising.

SYNTHETIC LIQUID FUELS DEMONSTRATION PLANTS

Coal-Hydrogenation Plant

Considerable information regarding early operating experience and plant improvements in the coal-hydrogenation demonstration plant has been published.31/ The following discussion covers additional tests and improvements to the plant, some of which have been described in more detail.32/ Part of the plant, including products storage and refinery equipment, is shown in figure 25. From June 27 through August 20, 1950, the unit was down for cleaning, mechanical repairs, and installation of six improved injection pump blocks.

Following the usual inert gas tests, hydrogen circulation was established for instrument testing and reactivation, and normal hydrogenation was started on August 21 on liquid-phase run No. 5, using Rock Springs, Wyo., coal. This was a combination 8,000 and 10,000 p.s.i. test that continued satisfactorily until September 10. At that time a leaky flange near the inlet to the first converter and a subsequent fire necessitated an emergency shut-down, 2 days before the normal shut-down planned for September 12, due to depletion of the supply of coal. During the 20-day test, approximately 1,130 tons of coal were converted to 143,000 gallons of oil, a yield of approximately 3 barrels per ton. The operation was carried out at 8,000 p.s.i. and 880° to 890° F. converter temperature except for the 4 days from August 27 through September 2, when the plant was operated at 10,000 p.s.i. with fair success. However, it was considered advisable to reduce pressure to improve injection-pump operation and to conserve the rapidly depleting stock of let-down valves. Another reason for the lower pressure was the desire to distribute the reaction between the two converters more equitably, because at the higher pressure most of the reaction took place in the first converter, and the second converter had a decided tendency to lose reaction, thus upsetting the smooth operation of the entire unit. As a result of this experience, plans were initiated to reduce the reaction volume by installing a liner in each converter.

In addition to the production of oils required for the vapor-phase manufacture of gasoline, the most important findings during the run were:

31/ Chaffee, C. C., and Clarke, E. A., Operating Experience with the Coal-Hydrogenation Demonstration Plant: Am. Soc. Mech. Eng. paper 50-PET-10, 1940; abs. in Mech. Eng., vol. 72, No. 12, December 1950, p. 1007.

32/ Synthetic Liquid Fuels. Annual Report of the Secretary of the Interior for 1950. Part I. Oil from Coal: Bureau of Mines Rept. of Investigations 4770, 1951, 74 pp.

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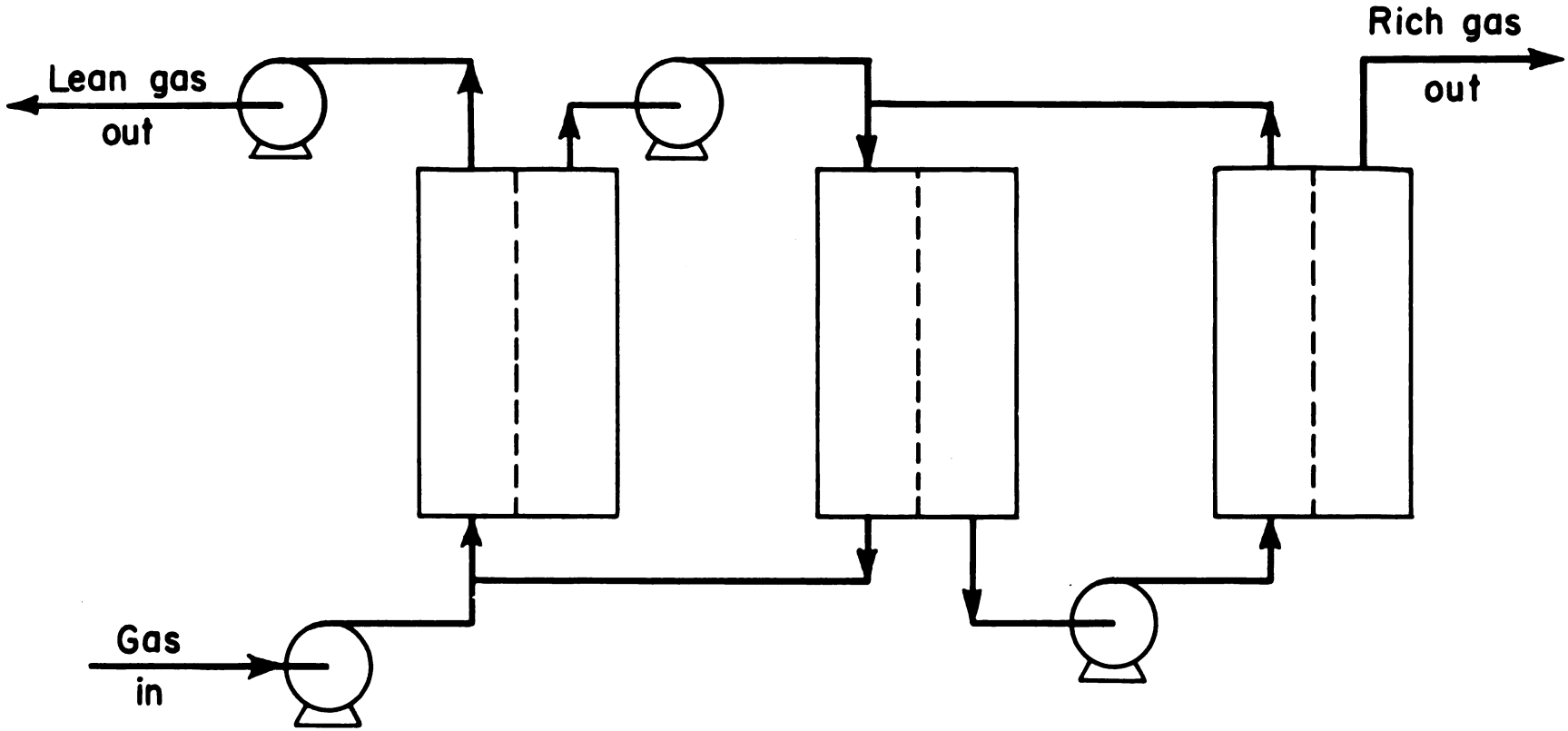

Figure 24. - Simplified flow diagram of a three-stage permeation process for the separation of gases.

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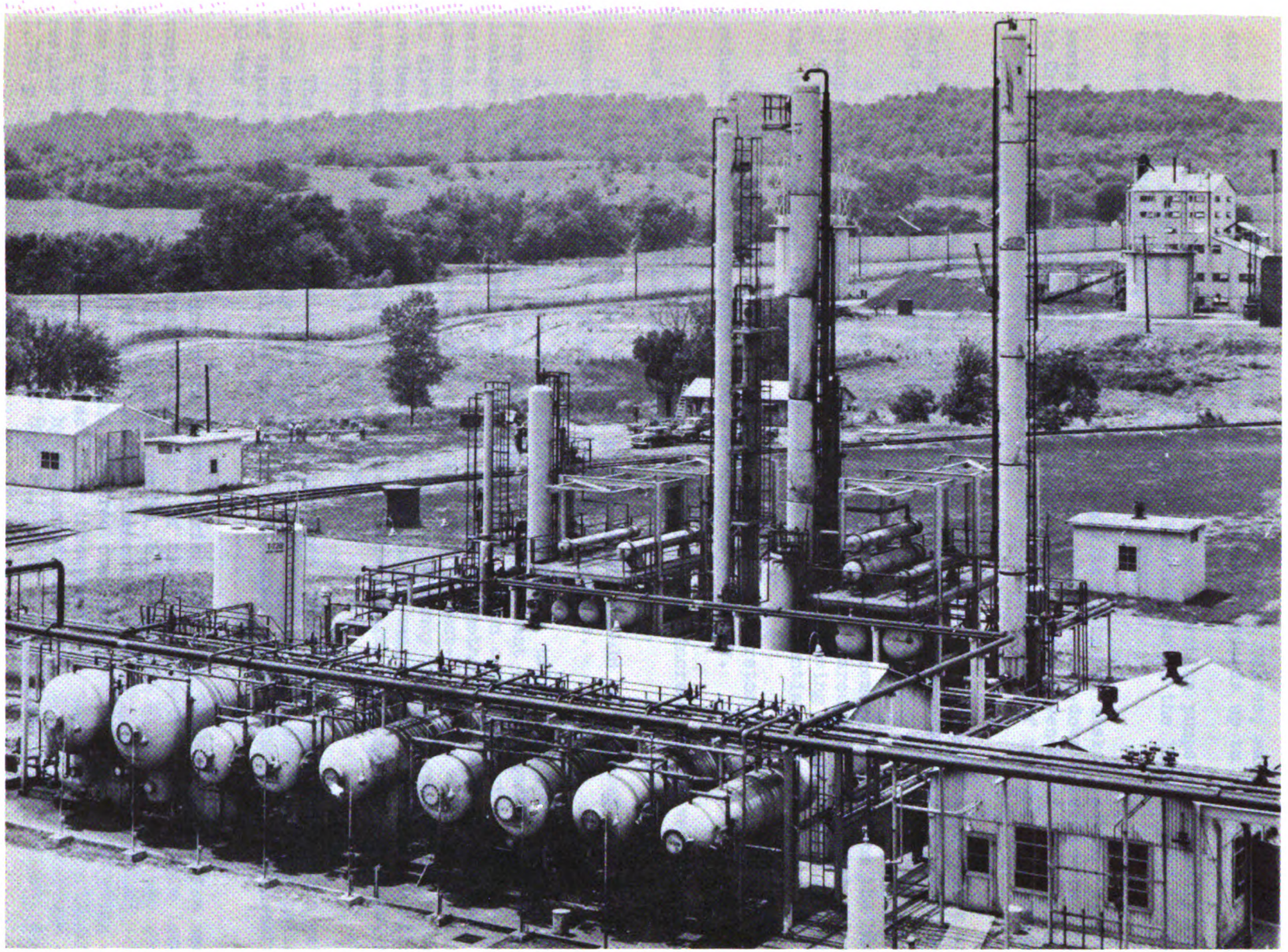

Figure 25. - Small modern refinery and storage vessels, Synthetic Fuels Demonstration Plant, Louisiana, Mo.

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1. The improved pump blocks, with exterior ball valves, satisfactorily pumped any of the hydrogenation injection oils or water at speeds up to 12 r.p.m.

2. The life of the chevron packing used in injection pumps was markedly improved by the addition of adequate, forced lubrication to the lantern ring and by the use of exterior drip lubrication to the plungers.

3. Owing to excessive breakage of stellite seats and disks, control-valve seats and disks in the high-pressure gas and clean-oil let-down service were changed to Cro-mo-van or chrome-molybdenum-vanadium alloy. Effectiveness of restricting orifices downstream from the control valves to absorb much of the wear and pressure drop was proved.

4. The duplex general service pump, equipped with hardened Madsen valves and Darcova cup plungers, continued to give excellent service in paste circulation at 250° C., but gave poor service at higher temperatures.

5. The horizontal, continuous centrifuge used to remove solids from the hot catchpot let-down continued to operate satisfactorily throughout the run at rates of 5 to 20 g.p.m. This apparatus was effective in removing a substantial part of the unconverted coal and ash in the oil without preferential removal of asphalt.

6. Readjustment of the tangential flow unit separator in the cold catchpot reduced the liquid carryover into the wash-oil scrubber.

7. Wash-oil scrubber operation was improved by increasing the vapor space below the packed section of the tower.

8. Wherever possible, flanged piping joints should be replaced by welded connections to eliminate potential sources of leakage.

A vapor-phase run (No. 2) was started October 5, using vapor-phase feed stock from Rock Springs, Wyo., coal, but the tar-acid content of the product indicated leakage, and the unit was shut down October 13. Upon completion of necessary repairs, production of 77 to 78 octane number (motor method), 8 to 10-pound vapor-pressure gasoline was resumed October 24. The product-feed heat exchanger continued to give considerable trouble, and there was a tendency for the preheater tubes to overheat, owing to the erratic operation of the exchanger and to the parallel flow arrangement of the preheater. Therefore, a new tubular exchanger was designed, and the preheater was changed to series flow.

The injection pumps equipped with improved blocks and better lubrication gave good service, and packing life increased to 15 days. Approximately 300,000 gallons of vapor-phase feed stock was converted to finished gasoline during the successful 30-day run.

After an extended winter shut-down, during which the liquid-phase system was completely cleaned and inspected and most of the indicated improvements were incorporated into the plant, liquid-phase run No. 6, using West Kentucky coal, was started March 30 and was completed May 17, after the entire coal supply was exhausted. During the run 2,160 tons of moisture- and ash-free coal was processed. All operating equipment continued to function satisfactorily throughout the run, and markedly improved results were obtained. This coal hydrogenated readily at a conversion pressure of 7,700 p.s.i. and a temperature of 875° F., using a 0.5 percent iron catalyst, at rates

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of 50 to 75 tons per day. All equipment in the stall and high-pressure area remained tight throughout the run. Despite high paste rates and considerable injection pump-valve trouble that developed as the run progressed, flow, pressure, and temperature conditions remained smooth and controllable throughout the run. The rather frequent difficulties with ball valves in the paste pumps have been traced to solid deposits in the suction lines. To eliminate these deposits, the lines have been redesigned. While the service life of most high-pressure control valves was excellent, the performance of heavy-oil let-down valves continued erratic, despite the installation of 3/32-inch restricting orifices immediately downstream from these valves. Hot catch-pot level control difficulties were accentuated by a tendency for reaction to start in the vessel, causing coke deposits. To alleviate these conditions, more agitation gas will be used.

Arrangements have been completed for a desander line to facilitate the removal of fine, sandy solids during operation. The flash-distillation system was operated about half of the time, usually with the Bird centrifuge, to reduce further the solids in the pasting oil, but mainly to control the asphalt level of the oil. To improve fume conditions and reduce manpower, a water-cooled metal conveyor for removal of solids will be obtained.

The vapor-phase charge from West Kentucky coal was fed to the unit for vapor-phase run No. 3 at an average rate of 12,740 gallons per day and was converted to an average rate of 12,713 gallons of gasoline per day. This above operation was performed smoothly at 10,000 p.s.i., with the inlet to the converter at 845° F. and with average bed temperature of 900° F. The over-all mechanical performance of the high-pressure plant equipment was adequate throughout the run. Likewise, all equipment in the distillation and gas-manufacturing areas remained satisfactory at all times.

At the end of this run, the following finished products were available for plant consumption and testing: 225,000 gallons of regular gasoline, 7,000 gallons of aviation base stock, 400 gallons of jet fuel, all from west Kentucky coal, and 60,000 gallons of regular gasoline produced from creosote-slop distillate. Samples of the first three items and gasoline from Rock Springs coal were sent to a commercial laboratory for complete tests.

A number of interesting process and mechanical improvements have contributed materially to make the present run more successful than previous operations. In the hydrogenation plant proper, the dependable operation of injection pumps and recycle compressors has allowed smooth flows to the unit at all times. The successful operation of the new, narrow range (800° - 900° F.) recording-temperature controller, regulating the flow of cooling gas to the preheater outlet line, held the converter entrance temperature within plus or minus 1°, which is essential for smooth control of bed temperatures. Improved operations in the distillation area resulted in supplying a well-blended feed stock.

Typical analytical and operational data of the liquid-phase operations are shown in tables 16 and 17.

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TABLE 16. - Typical analytical data, liquid-phase hydrogenation of west Kentucky coal

	Coal paste	Pasting oil	H.O.L.D.	Gasoline	Naphtha	Middle oil	Light oil bottoms	Cold catchpot product
Distillation, °F.:								
I.B.P.	-	475	360	116	210	242	592	190
5 percent	-	560	550	158	312	434	622	240
10 percent	-	604	606	194	360	464	638	297
20 percent	-	636	646	224	378	494	654	390
50 percent	-	-	-	270	402	534	686	593
70 percent	-	-	-	296	418	552	712	668
90 percent	-	-	-	334	446	578	-	731
E.P.	-	680	680	364	516	610	760	750
Recovery, percent	-	49.	31.	96.2	99.	99.	87.	93.5
Gravity	1.204	1.117	1.25	47.0	17.1	12.0	1.06	11.2
C_6H_6 - P. E. insolubles	41.3	8.4	25.2	-	-	-	-	-
C_6H_6 insolubles	43.7	5.5	19.0	-	-	-	-	-
Tar acids	-	-	-	4.1	38.0	22.0	-	-
Tar bases	-	-	-	2.5	.9	.1	-	-

Coal analysis

Proximate (moisture-free basis)

	Weight, percent
Moisture	-
Volatile matter	38.9
Fixed carbon	53.9
Ash	7.2

Ultimate

	Weight, percent
Ash	7.2
Sulfur	1.4
Hydrogen	5.2
Carbon	74.5
Nitrogen	1.4
Oxygen	10.3

Screen

	Weight, percent
On 35	0.2
Through 35, on 60	1.4
Through 60, on 100	5.7
Through 100, on 200	22.0
Through 200, on 230	10.1
Through 230	60.6

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TABLE 17. - Operational data, liquid-phase runs for fiscal year 1950-51; selected periods representative of lined-out operations

	Run 5		Run 6	
	Rock Springs (Wyo.) coal		West Kentucky coal	
	Period 1	Period 2	Period 2	Period 5
Pressure, p.s.i.g.	10,200	8,100	8,300	8,300
Recycle hydrogen, percent H_2	80	81	81.4	81.0
Paste injection, gal./day	30,800	29,100	25,000	31,000
Paste oil, gal./day	22,400	21,600	15,582	18,736
Coal, moisture-free, tons/day	56	56	54.9	67.5
Coal, moisture-free, weight percent	33.5	34.2	42.8	42.8
Catalyst, type	Tin oxalate	Tin oxalate	SnC_2O_4 - $FeSO_4$	Ferrous sulfate
Weight percent on moisture-free coal	0.1	0.1	0.04 0.8	2.5
Naphtha injection, gal./day	7,300	8,700	3,982	4,781
Wash oil injection, gal./day	14,500	18,500	40,500	45,100
Gas flows, cu.ft./day:				
Make-up hydrogen gas	3,960,000	4,150,000	2,215,000	2,287,000
Total hydrogen to stalls	10,900,000	10,200,000	9,420,000	9,031,000
Compressor recycle	12,300,000	13,100,000	11,900,000	12,400,000
Total paste gas	7,200,000	7,030,000	6,112,000	5,650,000
Cold paste gas	1,200,000	1,230,000	1,260,000	1,306,000
Hot paste gas	6,000,000	5,800,000	4,538,000	4,298,000
Total cooling gas	2,830,000	2,470,000	2,865,000	2,393,000
Hot catchpot agitation	890,000	700,000	628,000	792,000
Purge gas	1,580,000	1,720,000	600,000	389,000
Temperatures, °F.:				
Paste, preheater, inlet	164	159	157	157
1st section outlet	464	462	486	443
Preheater outlet	852	853	832	822
First converter, °F.:				
Reaction, top zone	874	878	858	856
Reaction, middle zone	865	854	871	872
Reaction, bottom zone	-	839	867	870
Second converter, °F :				
Reaction, top zone	874	893	862	859
Reaction, middle zone	874	892	870	869
Reaction, bottom zone	866	877	866	866
Temperatures, °F.:				
Hot catchpot, vapors	851	798	822	838
Hot catchpot, liquid (average)	688	690	637	634
Cold catchpot, liquid	187	190	193	195
Products from hydro, gal./day (based on run-down quantities):				
Heavy oil let-down	12,000	11,100	9,638	10,864
Cold catchpot, product-net	16,800	16,900	15,310	18,050
Gasoline	2,300	2,200	2,080	2,408
Naphtha	-	-	1,525	2,585
Middle oil	5,530	5,500	3,785	2,840
Flushing oil	510	-	-	-
Light oil bottoms	8,470	9,200	7,920	10,217
Total vapor-phase charging stock (recovery), bbl./ton of coal	3.3	3.3	3.44	3.07
Gasification (C_4 and lighter):				
Cu.ft./day	402,000	390,000	220,200	259,400
Weight percent on moisture- and ash-free coal consumed	26.7	26.4	13.6	13.3

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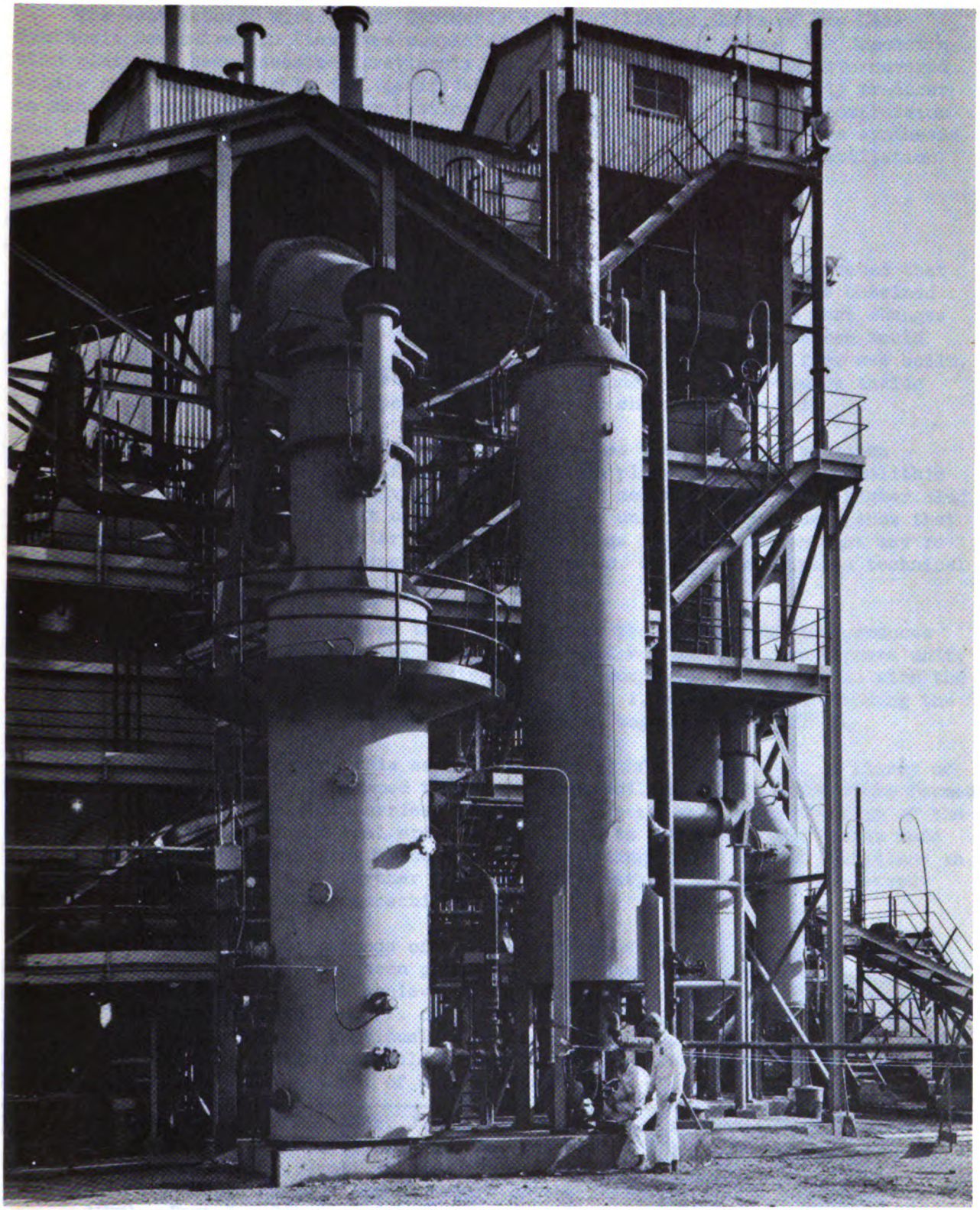

Figure 26. - Morgantown-type vertical coal gasifier and steam-coal preheater, Gas-Synthesis Demonstration Plant, Louisiana, Mo.

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Gas-Synthesis Plant

The contractors' work on the Synthesis and Distillation Units of the Gas-Synthesis Demonstration Plant was completed during the fiscal year. The demonstration plant has been described previously.33/34/ The plant operations have covered trials on a new, vertical, coal-gasification unit, the use of the Kerpely producer to manufacture synthesis gas for testing, the initial operation of the compression and purification units, the production and reduction of catalysts for the synthesis unit, and the testing and preliminary operation of the synthesis and distillation units.

Vertical Gasifier

The trial runs on the Koppers gasification unit made last year indicated that economy of operation would be improved if higher conversion of steam was obtained by reaction with the carbon of the coal. There was reason to believe that changes in the shape of the unit and the method of feeding coal, oxygen, and steam would result in increased conversion. In order to explore this, a new gasifier was built, which consisted of a vertical, refractory-lined cylinder, 4-feet 4-inches inside diameter, and approximately 20 feet high. Figure 26 shows the new unit.

The refractory chosen for the gasifier lining was a high purity aluminum oxide, ram-packed against inside forms, and fired in place to produce a monolithic lining. This material has the advantage of a higher melting point and a higher slag resistance than the silica brick used in the Koppers unit. If tests here show that monolithic linings are satisfactory, important savings in construction cost may result. Under a cooperative agreement, the Aluminum Co. of America supplied technical assistance and much of the material.

Feed materials are introduced tangentially near the bottom, and the products flow out the top to the same gas-handling system that was used for the Koppers unit. Provision has been made for either feeding the coal suspended in oxygen, as with the Koppers unit, with steam entering separately at about 1,000° F., or for feeding the oxygen separately, with the coal and steam preheated together to 1,000° F.

A unit was built to test this second, new method of feeding coal. A slurry of pulverized coal and water is pumped through a fired-coil preheater. The steam formed in the first part of the coil carries the coal in suspension through the rest of the coil in which both steam and coal are heated to 900° to 1,000° F. The trials made to date on this unit have given trouble with fluctuating flow rate and variations in the steam-coal proportions. Further tests are necessary to determine if a steady flow of a uniform mixture can be achieved.

There have been eight periods of test operation of the new gasifier, one of 30 hours' duration. Results have been promising, and in one test practically complete gasification of the carbon was achieved. In this case the requirements of 30.5 pounds of coal and 354 std. cu. ft. of oxygen per 1,000 cu. ft. of synthesis gas were lower than in any work previously done here. However, to date the tests have also shown that many difficult problems remain to be solved. Among these are very high local temperatures, removal of slag, and others of a similar nature.

33/ Dressler, R. G., and Bircher, J. R., The Bureau of Mines Gas-Synthesis Demonstration Plant at Louisiana, Mo. Am. Soc. Mech. Eng. paper 50-PET-9, 1950; abs. in Mech. Eng., vol. 72, No. 12, December 1950, p. 1007.

34/ Synthetic Liquid Fuels. Annual Report of the Secretary of the Interior for 1950. Part I. Oil from Coal: Bureau of Mines Rept. of Investigations 4770, 1951, 74 pp.

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Pebble-Type Steam Superheater

In the course of the previous operation of the Koppers gasification unit, a study was made of the performance of a circulating pebble-type steam superheater, with flow rates of 1,400 to 2,300 pounds per hour of steam superheated to 1,600° to 2,300° F.35/ Thermal efficiencies were calculated for different conditions, and data were presented on the rate of wear of the two types of pebbles used. Loss due to pebble wear amounts to about one-half pound per million B.t.u. transferred to the steam.

Kerpely Gas Producer

At the conclusion of tests of the Kerpely producer using oxygen and oxygen-enriched air blast, the results were analyzed and compared with similar operations previously reported.36/37/ The performance of the unit was quite satisfactory. It appears that the ultimate capacity of the producer is well above the capacity of the gas-handling system and of the oxygen plant. The raw-material requirements per M cu. ft. of CO + H_2 appear to be slightly lower for this unit than those achieved elsewhere.

Gas-Purification Unit

The adequate purification of the synthesis gas has been considered to be one of the major potential problems of the Fischer-Tropsch process. In addition to the substantially complete removal of sulfur compounds, it recently developed that the removal of the bulk of the carbon dioxide was highly desirable. Plans at Louisiana were modified accordingly by the substitution of diethanolamine for the triethanolamine previously chosen as a scrubbing medium, and the whole purification unit was put in preliminary operation. The first results have indicated that the removal of the necessary CO_2 in existing equipment is feasible and that a purified gas can be obtained containing appreciably less than 0.1 grain of total sulfur per 100 cu. ft. of gas, the figure set as a safe maximum for the Fischer-Tropsch catalyst. The purification unit is shown in figure 27.

The amine scrubbing system will reduce the CO_2 from 16 percent inlet to about 2 percent outlet and the H_2S from 130 grains per 100 cu. ft. to 10 to 15 grains. The balance of the H_2S is removed by passage through iron oxide on wood shavings and the organic sulfur by adsorption on active carbon. As a final insurance, the gas is then passed through beds of hot iron-oxide soda ash mixture, in case any organic sulfur passes the carbon beds.

Synthesis Reactor and Distillation Unit

In the course of testing operations of the synthesis and distillation units some modifications were found desirable. The most important change in the synthesis

35/ Batchelder, H. R., and Ingols, H. A., Performance of a Pebble-Heater-Type Steam Superheater: Bureau of Mines Rept. of Investigations 4781, 1951, 8 pp.

36/ Batchelder, H. R., Dressler, R. G., Tenney, R. F., Kruger, R. E., and Segur, R. D., Operation of Kerpely Producer with Oxygen-Enriched Blast: Proc. Am. Gas Assoc., 1950, pp. 348-354.

37/ Batchelder, H. R., Dressler, R. G., Tenney, R. F., Skinner, L. C., and Hirst, L. L., The Role of Oxygen in the Production of Synthetic Liquid Fuels from Coal: Bureau of Mines Rept. of Investigations 4775, 1951, 15 pp.

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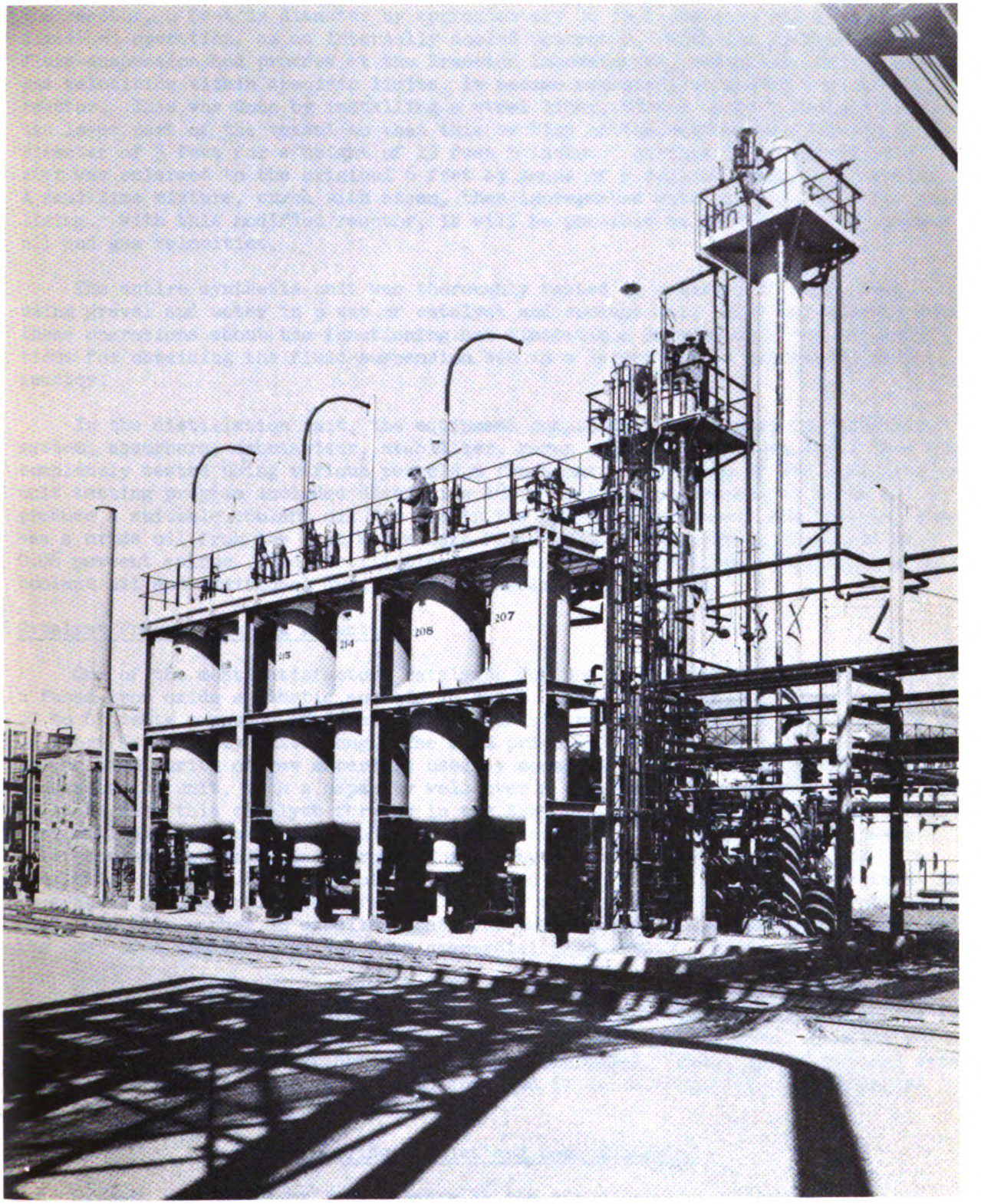

Figure 27. - Gas-purification unit, Synthetic Fuels Demonstration Plant, Louisiana, Mo.

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reactor was to adapt it for "jiggling" or fluid-suspension bed operation. Originally the reactor, 6 feet in diameter by approximately 30 feet overall, was designed for fixed-bed operation, as an internally cooled converter. With the development of the fluis-suspension bed process at the Bruceton laboratories, requiring coolant oil and gas velocities within specific limits, it became necessary to modify the synthesis reactor. This was done by installing a steel liner, backed up by a ceramic mass in the lower part of the vessel so that this section of the reactor was reduced to a diameter of 3 feet for a height of 19 feet 5 inches. At this point the reactor diameter was enlarged to the original 6 feet by means of a conical transition section. A sand-lime mixture, cured with steam, then impregnated with oil, was used for this lining. With this modified reactor, it will be possible to operate at the optimum oil and gas velocities.

The entire synthesis unit was thoroughly tested in a series of dummy runs, using gravel and water in place of catalyst and coolant oil. Much was learned from these operations about the functioning and limitations of equipment and the conditions for obtaining the fluid-suspension bed in a larger than a laboratory-size reactor.

In the distillation unit, the equipment comprising the primary fractionating system, absorbers, debutanizer, stabilizer, rerun column, and vacuum still have been completely tested using various petroleum fractions. One phase of the distillation unit testing program included distilling 18,500 gallons of low-sulfur crude oil to produce a suitable coolant oil for use in the synthesis reactor. The material chosen was a crude oil from the East White Lake Field in Louisiana, which contained only 0.06 percent sulfur. The distillation of this crude oil yielded 8,000 gallons of coolant oil and additional quantities of gasoline and naphtha.

Catalyst Preparation and Reduction

One of the most satisfactory catalysts for the fluid-suspension bed process is a fused-iron oxide synthetic ammonia catalyst, which is available commercially at 57 to 63 cents a pound. However, the number of producers is small, and the time of delivery is normally quite long. The high price and long delivery are due in part, to the high purity of raw materials used by commercial catalyst manufacturers. A manufacturing unit, with a capacity well over 2 tons of sized catalyst per day, was built to make this catalyst for use in the Bureau's operations. In this unit, ordinary rolling mill scale is blended with the desired promoters and fused between water-cooled electrodes in a trough furnace by means of alternating current. On completion of the fusion, the pig is permitted to cool and is then crushed to 8- to 16-mesh in jaw and roll crushers, followed by sizing in conventional screening equipment. In this manner an excellent catalyst was produced at approximately one-fourth the cost of a similar catalyst from commercial producers.

A catalyst reduction unit was designed to reduce 1 ton of fused catalyst per day. Some difficulties in operation, due to high carbon monoxide concentrations in the hydrogen gas available for reduction, were completely eliminated by using a nickel methanation catalyst before the reduction vessel. Tests on the reduced catalyst indicated a high degree of reduction, and it is believed that this material will perform well in the synthesis unit.

Engineering Studies and Cost Estimates

Process and equipment improvements in the demonstration plants have so progressed that space time yields assumed for commercial-scale plant estimates can be

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supported by operational data. Related industries and equipment manufacturers have continued to furnish reliable cost figures on the various plant components required in large-scale operations. The approach and methods used in synthetic fuel cost estimated were described in detail, with several examples.38/ The Bureau of Mines synthetic fuel cost estimates are being reviewed by a Subcommittee of the National Petroleum Council. Under the detailed scrutiny of this procedure, valuable technical and economical information is being exchanged between Bureau and industrial representatives.

A cost estimate of high-pressure residual oil hydrogenation was presented in condensed form.39/ Based on residual feed stock ranging from 1/2 to 2 dollars per barrel, figure 28 shows the predicted pay-out time for a 10,000 p.s.i. residual oil-hydrogenation plant based on prevailing product prices in effect at the time of publication.

A survey was made to determine the investment cost, steel requirements, manpower requirements, time to construct, and cost of construction of two, 15,000-barrel-per-day coal-hydrogenation plants. Each of these plants would produce:

	Barrels per calendar day
Phenols	620
Benzene	1,110
Aromatic blending stock	1/ 4,100
Motor gasoline	3,210
Butane and propane (LP-gas)	4,080

1/ Consists of 85 to 90 percent toluene and xylene.

Each plant was estimated to cost 163 million dollars, including 6-million-dollar interest during construction; approximately 100,000 tons of steel would be required, and approximately 14 million man-hours of construction labor would be required for each plant.

A design and cost estimate was made of a hydrocarbon combustor unit for hydrogen production in a 30,000-barrel-per-day hydrogenation plant. Oxygen requirement for the combustor was calculated to be 827.5 M std. c.f.h. of 95 percent purity, steam requirement in the shift reactors to be 214,000 pounds per hour. This unit could be used to replace a 6-unit pressurized pebble heater plant described previously.40/

A rough cost estimate was made for a Fischer-Tropsch plant using air instead of oxygen to gasify the coal. It was found that the capital cost of a 10,000-barrel-per-day plant using air is 23 million dollars greater and that the plant would consume 1,388 tons per day more coal because the heating and power requirements would be greater.

38/ Skinner, L C., Preparation of Synthetic-Fuel Cost Estimates by the Coal-to-Oil Demonstration Branch: Am. Soc. Mech. Eng. paper 50-PET-11, 1950; Abs. in Mech. Eng., vol. 72, No. 12, December 1950, pp. 1007-1008.

39/ Skinner, L. C., Donath, E. E., Schappert, H., and Frese, E., Residual Oil-Hydrogenation: Petrol. Refiner, vol. 29, July 1950, pp. 83-87.

40/ Hirst, L. L., Markovits, J. A., Skinner, L. C., Dougherty, R. W., and Donath, E. E. Estimated Plant and Operating Costs for Producing Gasoline by Coal Hydrogenation: Bureau of Mines Rept. of Investigations 4564, 1949, 83 pp.

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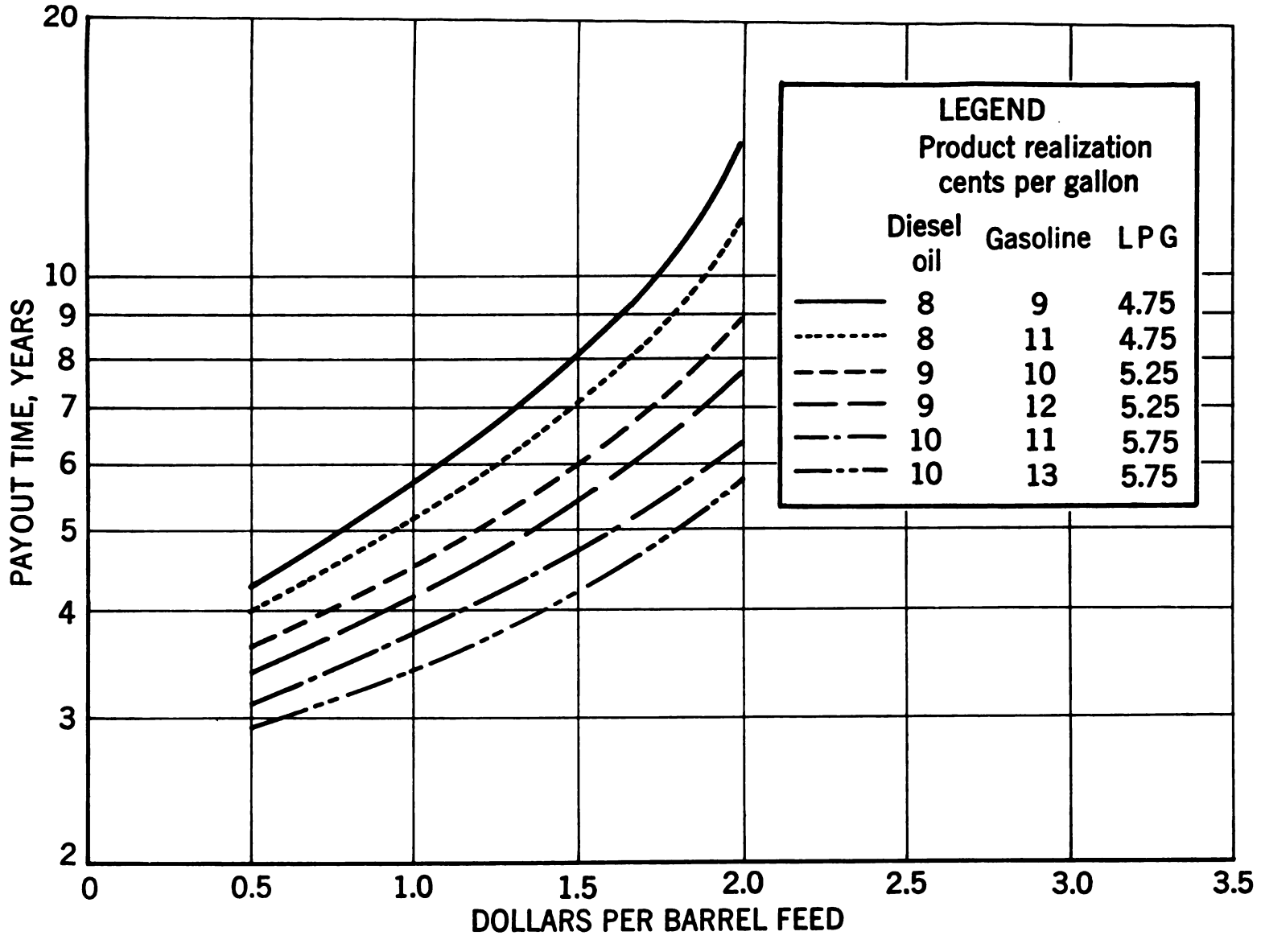

Figure 28. - Predicted payout time for a 10,000 p.s.i. residual oil-hydrogenation plant for various cost and realization values.

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The possibilities of ammonia as a byproduct from a gas-synthesis plant was investigated. It was found that the potential ammonia production from the tail gas of a 10,000-barrel-per-day plant would be about 1,283,000,000 pounds per year, and, therefore, it would not find a ready market. Natural gas is a much cheaper raw material for ammonia production, because the tail gas from a gas-synthesis plant contains appreciable carbon dioxide, which must be removed for ammonia synthesis.

A preliminary cost estimate was prepared for producing 3,600,000 std. c.f.h. of gas by atmospheric gasification of Pittsburgh seam coal for use in an existing ammonia plant as raw ammonia synthesis gas, raw methanol synthesis gas, or as a combination of the two. Production of 650 tons per day of ammonia on four lines would require 540 tons per day of oxygen and 868.8 tons per day of coal. Production of ammonia on one line (162 tons per day) and methanol on three lines (150,000 gallons per day) would require 850 tons per day of oxygen and 1,032 tons per day of coal. The investment required, using some existing equipment, would be $12,900,000, which gives a cost of raw synthesis gas of 13.2 cents per 1,000 cu. ft. This includes amortization of 6-2/3 percent per year, but no profit or taxes.

A study was made to determine the quantity of sulfur that can be recovered from one 15,000-barrel-per-day, coal-hydrogenation plant requiring 225 tons per hour of "as mined" western Kentucky coal. The study indicated that 58.2 tons per day of free sulfur could be recovered. In addition to the free sulfur, 203 tons per day of ammonium sulfate and refuse material containing 175 tons per day of pyrites could be recovered. To produce the ammonium sulfate, 180 tons per day of sulfuric acid would be required, containing 58.7 tons per day of sulfur, which is almost equal to the total free sulfur produced. Thus, the net sulfur produced would be in the form of ammonium sulfate.

Other engineering and cost estimate studies included: Estimation of high-pressure tubing requirements for a coal-hydrogenation plant; estimation of utility (power, water, and steam) requirements for commercial-scale production of hydrogen by the hydrocarbon steam cracking process for use in shale oil hydrogenation; and comparison of operating costs for atmospheric and pressure gasification, for which data are shown in table 18.

SYNTHETIC LIQUID FUELS PUBLICATIONS

Books

A book on the Fischer-Tropsch and related syntheses has been published[41] as the result of an extensive and critical review of the large number of reports and documents pertaining to these processes. In addition, much information on thermodynamics and on hetergeneous catalysis is included.

A resumé of synthetic liquid fuels processes, previously published in the technical press, was incorporated in a book on chemical processes.[42]

41/ Storch, H. H., Golumbic, N., and Anderson, R. B., The Fischer-Tropsch and Related Synthesis, Including a Summary of Theoretical and Applied Contact Catalysis: John Wiley & Sons, Inc., New York; Chapman & Hall, Ltd., London, April 1951, 610 pp.

42/ Kastens, Merritt L., in collaboration with Hirst, L. L., and Chaffee, C. C., Liquid Fuel from Coal: Ind. Eng. Chem., vol. 41, No. 5, May 1949, pp. 870-885; vol. 42, No. 8, August 1950, p. 1607; Modern Chemical Processes (by Editors of Ind. Eng. Chem.), Reinhold Pub. Corp., 1950, pp. 56-71.

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TABLE 18. - Operating cost of atmospheric vs. pressure gasification for a 10,000-bbl./day gas-synthesis plant

	Gasification, atmospheric			Gasification, 450 p.s.i.g.		
	Capital cost	Operating hp.	Daily operating cost	Capital cost	Operating hp.	Daily operating cost
Unit:						
Coal preparation.....	1/ $4,734,000	7,500	$4,357	1/ $4,734,000	7,500	$4,357
Gasifier.....	2/ 15,300,000	6/ (66,800)	1,067	3/ 10,700,000	6/ (58,500)	6/ (1,913)
Oxygen.....	4/ 17,600,000	70,800	18,921	4/ 17,600,000	70,800	18,921
Oxygen compression.....	300,000	2,600	450	3,700,000	22,600	5,570
Synthesis-gas compression...	5/ 14,035,000	101,155	21,108	-	-	-
Coal.....246 ton/hr. @ $2.75 ton	-	-	16,236	-	-	16,236
Total.....	7/ $51,969,000	115,255	$62,139	7/ $36,734,000	42,400	$43,171
Cost of synthesis gas to purification....¢/1,000 cu. ft.	-	-	15.00	-	-	10.40
Cost of synthesis gas to purification.....¢/gal. of hydrocarbon product	-	-	13.50	-	-	9.40

1/ Obtained from a proposal from Kennedy-Van Saun Manufacturing & Engineering Corp., Apr. 5, 1951.
2/ Obtained from N.P.C. atmospheric-pressure gasification design, letter Gaucher to Hirst, Dec. 18, 1950. A preliminary capital cost figure from N.P.C. of $3,550,000 total direct cost, for 3 units. 9 are required.
3/ Bureau of Mines pressure gasification unit with some adjustments in design to make comparable with N.P.C. design. Bureau's cost figures. Letter, Hirst to Carlsmith, May 2, 1951.
4/ Capital cost and power from N.P.C. letter Carlsmith to Hirst, April 5, 1951.
5/ Capital cost and operating costs from Clark bid of Aug. 25, 1950, as per letter, Skinner to Schmidt, April 9, 1951.
6/ Parentheses indicate credit.
7/ Total does not include 3 percent interest and 5 percent working capital.

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Reviews and Abstracts

The current (1949-50) literature on fluid dynamics was summarized in a review,[43] which also included references to earlier material in foreign journals. A review on the oxo synthesis and its modifications[44] discussed postwar developments of this reaction. Research on synthetic fuels by the Bureau of Mines has been outlined for an Office of Technical Services publication.[45]

Compilation of Synthetic Liquid Fuels Abstracts has been continued bimonthly to give a comprehensive coverage of current literature and patents.

Bibliographies

Part I of a bibliography of pressure hydrogenation has been published.[46] This publication is a review and compilation of technical literature dealing with the history, development, and commercial application of the Bergius and related processes for the hydrogenation, under pressures greater than atmospheric, of liquid and solid carbonaceous materials, including coal, lignite, their distillation and extraction products, pitches and tars, and petroleum and its distillation residues. A bibliography of the Fischer-Tropsch reaction and related processes has been compiled and is being prepared for publication.

Foreign Documents

Several foreign documents in the field of synthetic fuels have been translated and published. These include a German publication[47] on basic calculations for designing converters; a discussion of German research on the isosynthesis,[48] describing the synthesis of branched-chain hydrocarbons; and Japanese reports on the synthesis of hydrocarbons from carbon monoxide and hydrogen,[49][50] including a description of process development.[51] About 500 more foreign documents have been indexed and are now being processed. An eight-volume German text on high-pressure hydrogenation was translated as a restricted document.

43/ Leva, M., and Weintraub, M., Fluid Dynamics: Ind. Eng. Chem., vol. 43, January 1951, pp. 90-99.

44/ Wender, I., Orchin, M., and Storch, H. H., New Modifications of the Oxo Process: Armed Forces Chem. Jour., vol. 4, October 1950, pp. 4-9.

45/ Cohn, E. M., Bureau of Mines Research on Synthetic-Liquid-Fuels Processes: Bibliography of Tech. Reports, U. S. Dept. of Commerce, Office of Tech. Services, vol. 15, January-June 1951, pp. 65-66.

46/ Wiley, J. L., and Anderson, H. C., Bibliography of Pressure Hydrogenation. I. Review and Compilation of the Literature on Pressure Hydrogenation of Liquid and Solid Carbonaceous Materials: Bureau of Mines Bull. 485, 1950, 306 pp.

47/ Wirth, Gustav. (translated by Grass, R. C. and Kandiner, H. J.), The Evaluation of Converters for Exothermic and Endothermic Catalytic Reactions Occurring Within Narrow Temperature Limits: Bureau of Mines Inf. Circ. 7587, 1950, 13 pp.

48/ Pichler, Helmut, and Ziesecke, Karl-Heinz, (translated by Brinkley, R., technical revision by Golumbic, N.), The Isosynthesis: Bureau of Mines Bull. 488, 1950, 39 pp.

49/ Tsutsumi, Shigeru. (revised and edited by Grass, R. C., translated by Technical Japanese Translation Service), The Synthesis of Hydrocarbons. (Report of the Imperial Fuel Research Institute of Japan, July 25, 1935): Bureau of Mines Inf. Circ. 7594, 1951, 60 pp.

50/ Watanabe, Shiro. (revised and edited by Grass, R. C., translated by Technical Japanese Translation Service), Bench-Scale Studies of the Fischer-Tropsch Synthesis over Iron, Nickel, and Nickel-Cobalt Catalysts (Japan): Bureau of Mines Inf. Circ. 7611, 1951, 26 pp.

51/ Kodama, S., Funabashi, W., Hashimoto, G., Hirao, T., Tahara, H., Matsumura, A., Kato, J., and Tarama, Y. (revised and edited by Grass, R. C., translated by Technical Japanese Translation Service), Process Development in the Hydrocarbon Synthesis to 1941: Bureau of Mines Inf. Circ. 7593, 1951, 41 pp.

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